Cryo-200
VASE◆ Cryostat Attachment

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The CRYO-200 option adapts a Janis Research model ST-400 SuperTran UHV cryostat to a GB-700 VASE ellipsometer base. This allows the user to control the sample temperature over an extremely wide range, from 4.2 Kelvin to 700 Kelvin, while making transmission measurements or VASE measurements. The windows on the cryostat have a limited acceptance angle, which in turn limits the VASE measurement angle of incidence to a range of 65° to 75°.

For a complete understanding of the functionality and operation of the cryostat and its support systems it is necessary for the user to read and understand all the manuals provided by Janis Research. This application note is a VASE operation supplement and is not a substitute for the training and experience needed for the correct and safe operation of a cryostat. PLEASE READ AND UNDERSTAND THE MANUALS!

Cryostat nomenclature can be found in the "Introduction to Laboratory Cryogenics" manual provided by Janis Research. Schematic diagrams located at the back of the "Operating Instructions for the Janis Research Supertran System" are also useful.

This application note assumes the reader (user) understands the operation of the VASE instrument (See VASE Hardware Manual and WVASE32 Software Manual).

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Section 1 Safety Concerns

User Safety

Operators and Observers MUST to be aware of these safety concerns:

1) **The evaporating gas from the cryogen (Helium or Nitrogen) is an asphyxiant. While it is not toxic, it will displace the oxygen in the room and will KILL you just the same! The system MUST be set up to be in a WELL VENTILATED area.**
2) **Use only INERT cryogens (e.g. Helium or Nitrogen). Do NOT USE liquid oxygen or hydrogen as the cryogen.**
3) **The cryogen and anything it has been in contact with is unimaginably cold and will cause SEVERE frostbite (cold burns). The primary danger is from items that have been in contact with the cryogen. They stay cold and must be allowed to warm up to room temperatures before being handled. ALWAYS wear long sleeve shirts, full length pants, covered toe shoes, safety glasses and insulated gloves when handling cryogen and items that have been in contact with cryogen!**

Equipment Safety

Operators NEED to be aware of these operational concerns:

1) **The transfer line MUST be removed from the cryostat for operation at temperatures above 475 Kelvin. The transfer line will be damaged if it is overheated.**

2) **The cryostat vacuum shell MUST be under vacuum and continuously pumped during cryostat operation. At elevated temperatures, outgassing will occur and atmospheric contaminants (oxygen) will corrode the sample mount. At low temperatures, atmospheric gases will condense (deposit) on the sample, altering the sample morphology on a continual basis making meaningful measurements of the sample difficult.**

3) **When the cryostat is mounted on the ellipsometer, do not move the system angle of incidence (AOI) beyond the 0° to 90° range, either manually or automatically. Doing so will result in damage to the flexible vacuum hose.**

4) **The cryostat exhaust port tends to build up condensation or frost at high cryogen flows. This condensation or frost should be wiped off as quickly at it is formed. Water and electrical equipment do not mix.**

Section 2 System Setup

Turbo Pump Setup

Any foreign matter inside the vacuum system will likely result in major damage to the pump!

Note: Cleanliness is absolutely essential in this procedure! Any foreign matter inside the vacuum system will likely result in major damage to the pump! Please read the instruction manuals for the Edwards Active Gauge Controller and the Edwards Turbomolucular pump and controller before performing this procedure. A schematic diagram located at the back of the "Operating Instructions for the Janis Research Supertran System" contains a general vacuum pump layout and useful nomenclature.

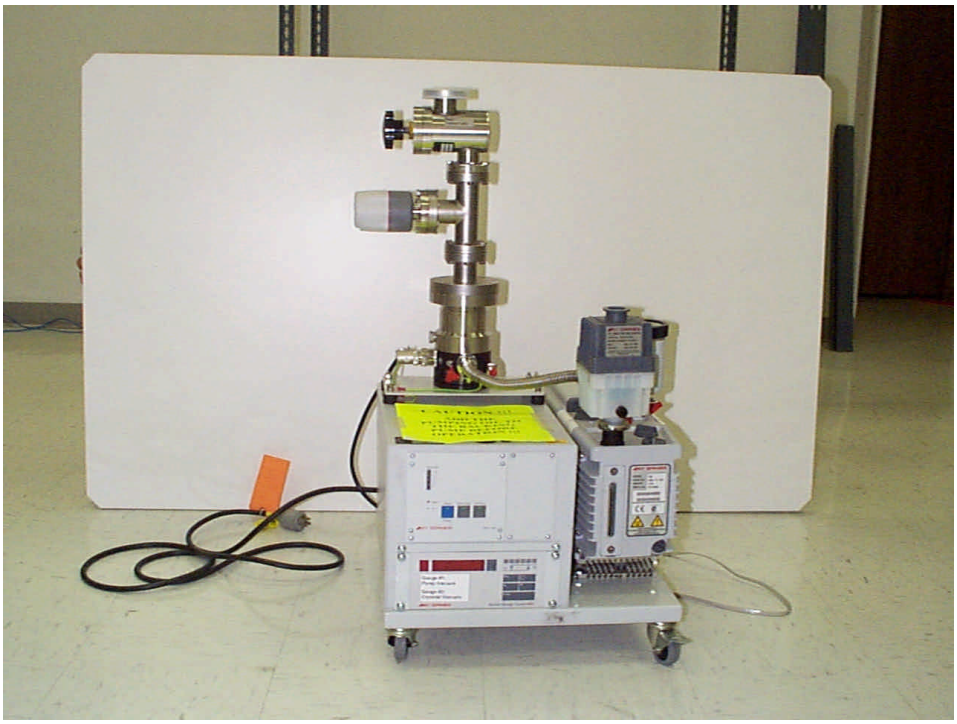

Figure 1

Tools Needed:

- Clean room gloves
- 1/2" combination wrench, 2 each

Parts Needed:

- Pumping Station, 1 each
- Pump Oil, ½ liter
- 2.51” ID copper gasket, 1 each
- Vacuum tee with gauge and valve, 1 each
- 5/16-24 bolts with nuts and washers, 8 each

Assembly Steps:

1. Fill the roughing pump with pump oil (Figure 2) as per specification in the pump manual. (Remove the caution notice when finished.)

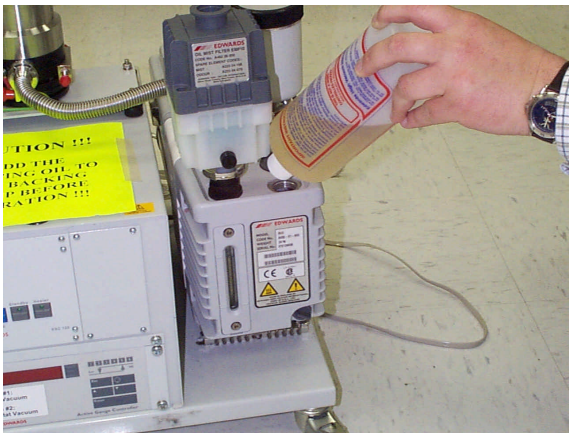

Figure 2

2. Install a new 2.51” ID copper gasket in the seal flange at the top of the turbomolecular pump (Figure 3).

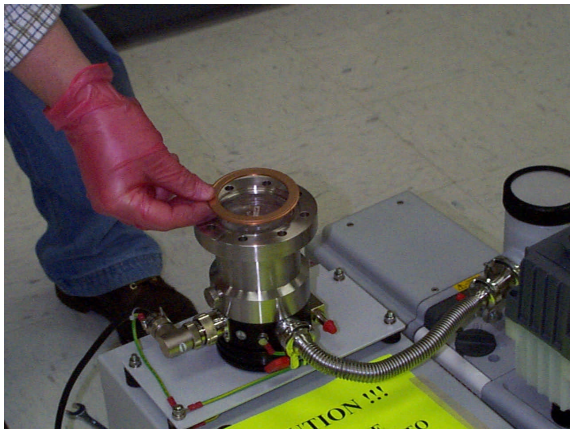

Figure 3

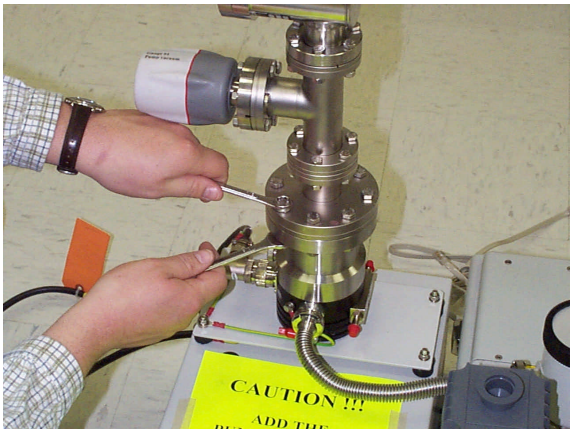

Figure 4

3. Place the vacuum tee on the seal flange at the top of the turbomolecular pump with the gauge and valve facing away from the roughing pump.
4. Install the eight 5/16-24 bolts, washers, and nuts finger tight, making sure the seal flanges are parallel.

Incrementally tighten bolts in cross-cross pattern to avoid leaks.

5. Using the two ½” combination wrenches (Figure 4), gradually tighten the bolts to 15 lb-ft in ¼ to ½ turn increments using an alternating criss-cross star pattern (the seal flanges should remain parallel throughout the entire bolt tightening sequence).

6. Connect Active Gauge Controller cable labeled “Pump Vacuum” to the gauge on the vacuum tee (Figure 5). The other will be used later on the cryostat gauge.

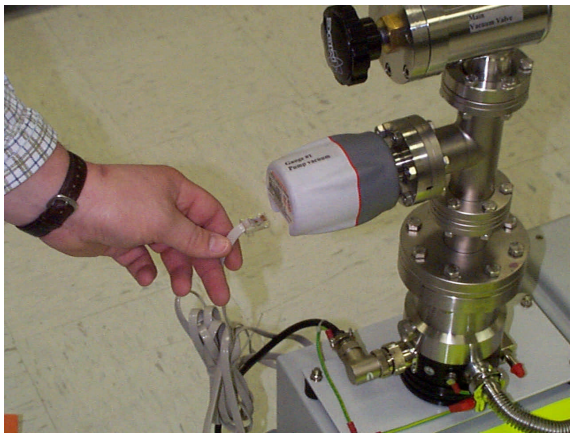

Figure 5

7. The system is now ready to test the integrity of the copper gasket seal just installed. (If the seal needs to be replaced, it is much more convenient to fix it before the full work table is assembled.)
8. Start this check by making sure the main vacuum valve is completely closed.
9. Connect the pumping station Isobar (power strip) to AC power.
10. Make sure the power switch for each of the pumping station components is in the ‘on’ position (turbomolecular pump controller, active gauge controller, and roughing pump) as well as the main power switch (all switches are located on the back side of the pumping station).
11. The pumping station should be powered and the displays active. Toggle the vacuum gauge display to read the pump vacuum (see the Edwards Active Gauge Controller manual).
12. Start the vacuum pump by pressing the ‘Start’ button. The pressure should quickly drop to below 10^{-4} Torr. If not, there is probably a leak in the seal between the turbomolecular pump and the vacuum tee. If there is a leak, try tightening the clamping bolts. If the leak persists, press the ‘Stop-Start’ button to stop the pump, and let the system return to atmospheric pressure. Replace the copper gasket seal and try pumping down again.
13. After the pressure reaches 10^{-5} Torr, press the ‘Stop-Start’ button to stop the pump, and let the system return to atmospheric pressure.

The pumping station is now ready for installation in the system.

For additional information on the pumping station components, refer to the appropriate manual (included with the system). There is no separate manual for the pumping station as a system.

Work Table Setup

Note: This procedure covers the setup of the CRYO-200 option as it relates to the ellipsometer and not the setup of the ellipsometer itself. Please read "Operating Instructions for the Janis Research SuperTran System" from Janis Research. Schematic diagrams at the end of the Janis manual contain a general cryostat layout and useful nomenclature. Also read "User's Manual Model 330 Autotuning Temperature Controller" from LakeShore. Section 2.6.2.2 contains useful thermocouple hook up information.

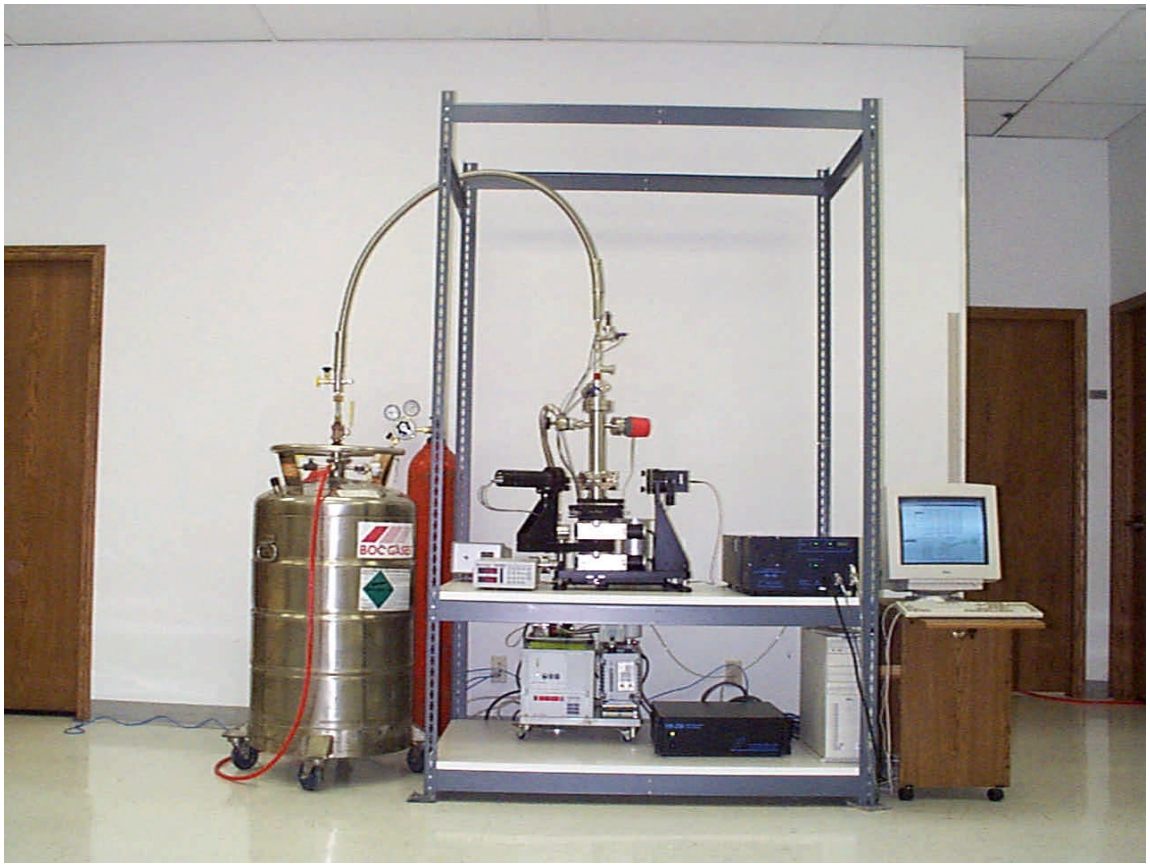

Figure 6

Tools Needed:

- Rubber mallet
- 4mm key wrench
- 7/16" combination wrench
- 9/64" key wrench
- Small straight blade screwdriver
- Small phillips tip screwdriver

Parts Needed (in addition to the ellipsometer):

- Table components:
  - 8' side rails, 4 each
  - 5' support rails, 6 each
  - 3' support rails, 6 each
  - 3' support brace, 1 each
  - Base, 1 each

    - Tabletop, 1 each

- Cryostat components:
    - CRYO-200 storage stand
    - CRYO-200 cryostat assembly
    - M5 by 16 mm socket head cap screws, 4 each
    - 1.45" ID copper gasket, 1 each
    - Flexible vacuum line, 1 each
    - ¼-28 bolts and nuts, 6 each
    - #8-32 Socket head cap screws, 6 each
    - #8-32 nut plate, 3 each
    - .64" ID copper gasket, 1 each
    - Cryogen transfer line, 1 each
    - Cryogen dewar (not included)

- Temperature controller components:
    - Temperature controller, 1 each
    - Power cord, 1 each
    - Cold Junction Compensator, 1 each
    - Thermocouple cable, 1 each
    - Heater cable, 1 each

- Exhaust port heater components:
    - Heater controller, 1 each
    - Power cord, 1 each
    - Heater cable, 1 each
    - Pumping station components:
    - Assembled pumping station, 1 each
    - Isobar power strip, 1 each

Facility Needs:

- Well ventilated room
- Computer Stand
- 4' by 8' floor space (plus room for computer stand) with 9' ceiling
- 15 amp 120 VAC outlet (separate circuit from the ellipsometer)

Assembly Steps:

1. Assemble the outer frame (Figure 7 and Figure 8). A rubber mallet will be necessary to persuade the supports into place. All four base supports (two 3', two 5') should be in the #2 and #3 holes from the bottom. Three of the top supports (one 3', two 5') should be in the #2 and #3 holes from the top. The last top support (3') should be in the #6 and #7 holes from the top (this acts as a support for the cryogen transfer line and should be located on the left hand side of the table).
2. Install the base shelf.

Figure 7

Figure 8

3. Place the vacuum pumping station on the base, located toward the back, left hand corner as in Figure 11. The front of the pumping station should be 17” from the front of the base. The left side of the pumping station should be 12” from the left side of the base.
4. Connect the pumping station to the Isobar power strip. There is a separate Isobar for the ellipsometer system.
5. Install the four table support rails in the#17 and #18 holes from the bottom (Figure 9).
6. Install the 3’ table-center cross brace with the vertical brace to the right of the mounting rivets. (Figure 11)

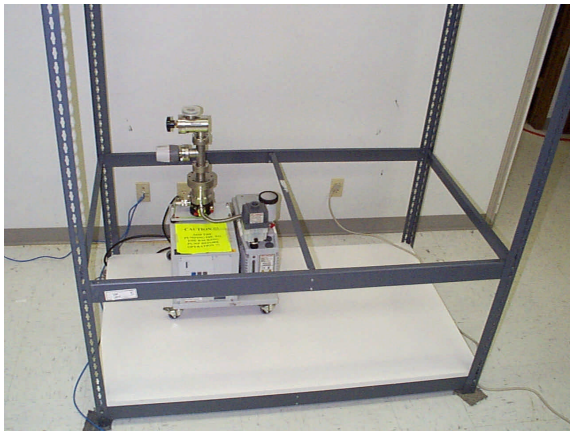

Figure 9

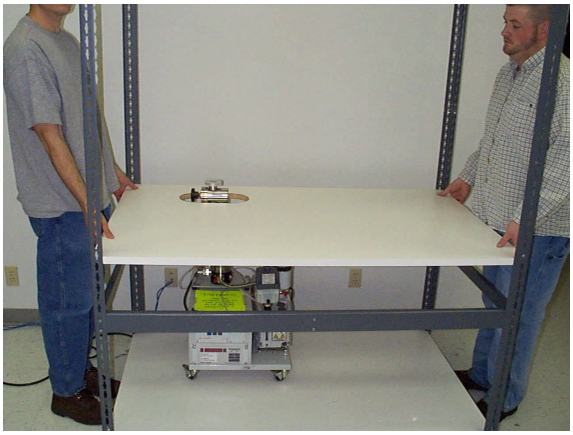

Figure 10

7. Making sure the main vacuum valve and pump vacuum gauge fits through the hole, carefully install the tabletop (Figure 10). Note the cross brace shown in Figure 11 should be installed for this step although it not depicted in Figure 10.
8. Place the ellipsometer base on the tabletop near the main vacuum valve (Figure 11). The front of the ellipsometer base should be 10” from the front of the tabletop. The left side of the ellipsometer base should be 16” from the left side of the tabletop.

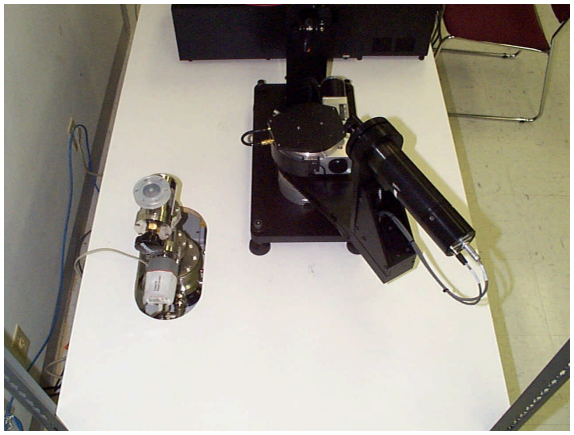

Figure 11

9. Carefully place the CRYO-200 assembly on the ellipsometer base (Figure 12), oriented such that the z-axis adjust knob faces toward the input unit when the AOI is at 0°.

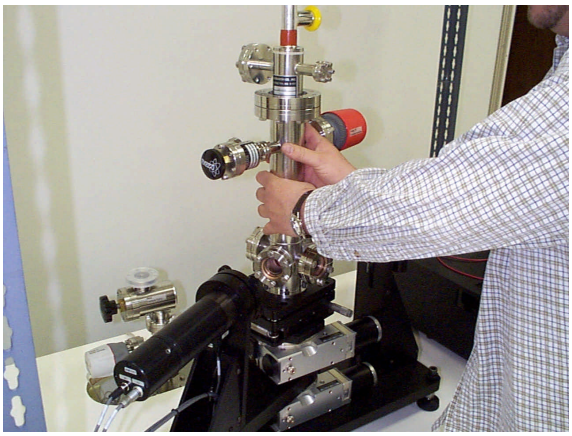

Figure 12

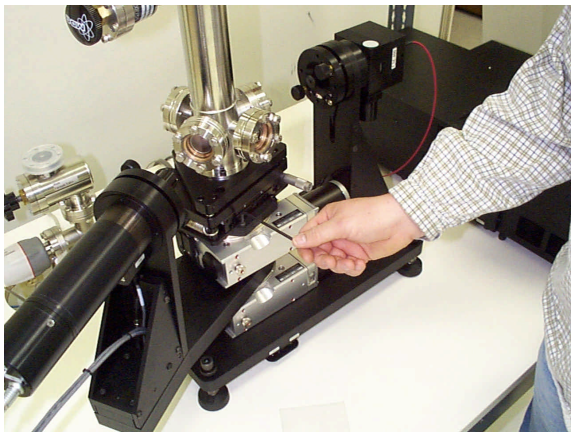

Figure 13

10. Install four M5 socket head cap screws finger tight to mount the CRYO-200 assembly to the ellipsometer base.
11. Use the 4mm key wrench to tighten the M5 socket head cap screws (Figure 13).
12. Install a new 1.45” ID copper gasket in the seal flange at the top of the main vacuum valve (Figure 14).
13. Mate the flexible vacuum hose 2-3/4” seal flange up to the main vacuum valve seal flange at the top of the vacuum tee.
14. Install the six ¼-28 bolts and nuts finger tight through the seal flanges, making sure the seal flanges are parallel.

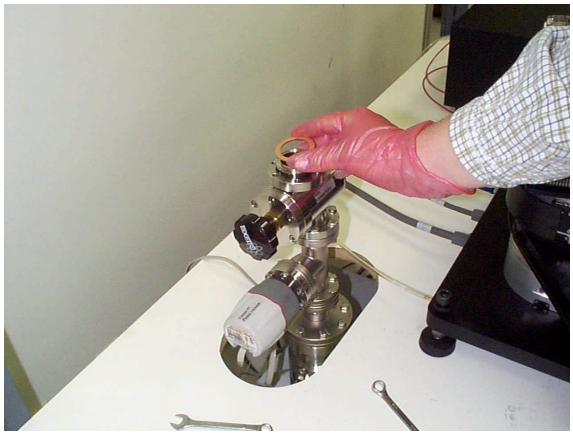

Figure 14

Figure 15

15. Using two 7/16” combination wrenches (Figure 15), gradually tighten the bolts to 12 lb-ft in ¼ to ½ turn increments using an alternating criss-cross star pattern (the seal flanges should remain parallel throughout the entire bolt tightening sequence).
16. Install a new .64” ID copper gasket in the seal flange of the cryostat vacuum valve (Figure 16).

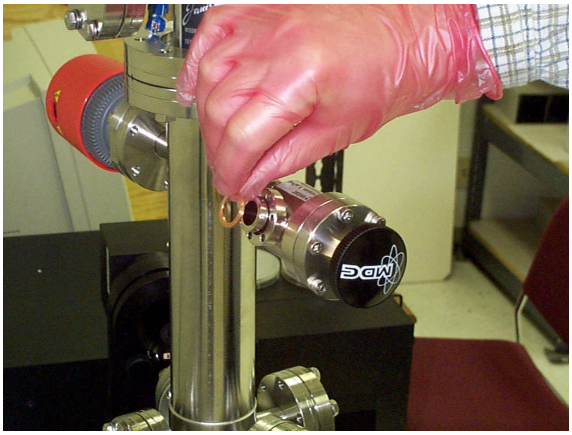

Figure 16

17. Mate the flexible vacuum hose 1-1/3” seal flange to the cryostat vacuum valve seal flange.
18. Install the six #8-32 screws and three nut plates finger tight through the seal flanges, making sure the seal flanges are parallel.
19. Make sure the AOI can move the full 0° to 90° range without placing undo strain on the flexible vacuum hose (refer to VASE Hardware manual for instructions on manually changing the AOI).
20. Using the 9/64” key wrench (Figure 17), gradually tighten the screws to 7 lb-ft in ¼ to ½ turn increments using an alternating criss-cross star pattern (the seal flanges should remain parallel throughout the entire bolt tightening sequence).

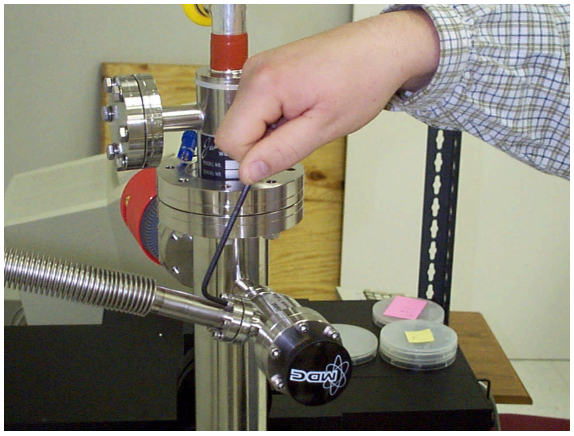

Figure 17

21. Place the exhaust port heater controller on the tabletop next to the main vacuum valve (Figure 18).

22. Place the temperature controller on the tabletop next to the ellipsometer base (Figure 18).

Figure 18

23. Connect the exhaust port heater to its controller using the cable provided (Figure 19 and Figure 20).

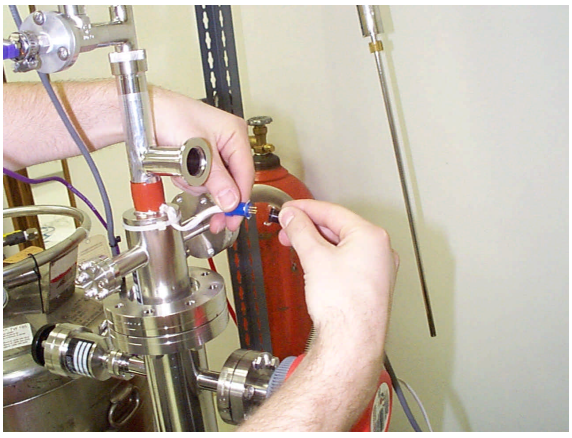

Figure 19

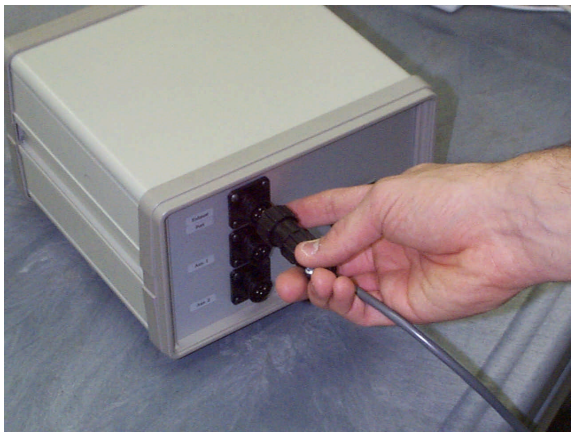

Figure 20

24. Connect the exhaust port heater controller to the vacuum pumping station Isobar using the cord provided.

25. Connect the cryostat thermocouple to the cold junction compensator using the cable provided (Figure 21 and Figure 22).

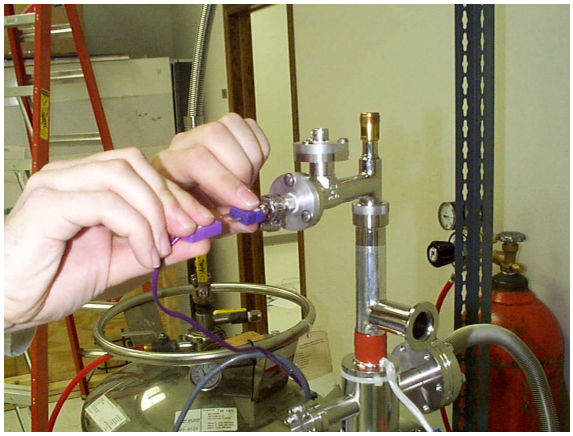

Figure 21

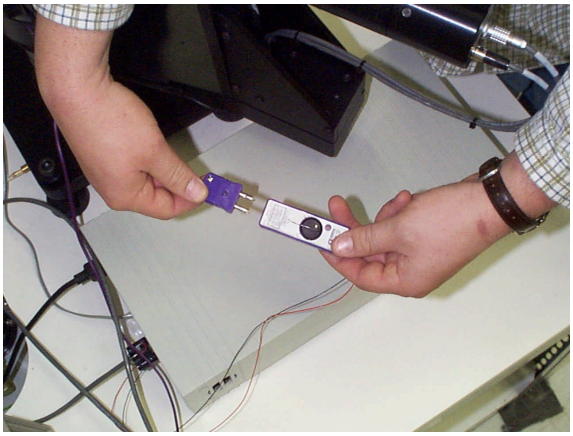

Figure 22

26. Using the small phillips tip screwdriver, connect the cold junction compensator to the temperature controller (Figure 23).

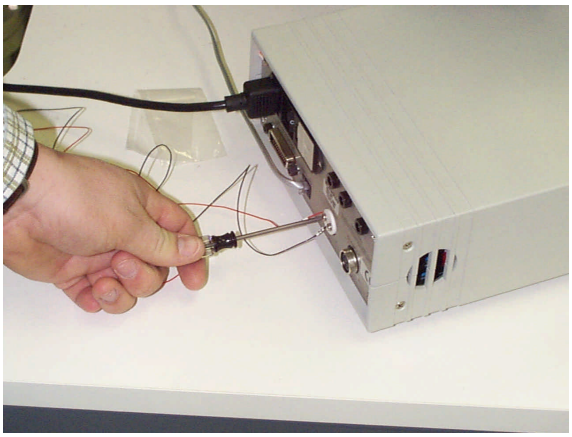

Figure 23

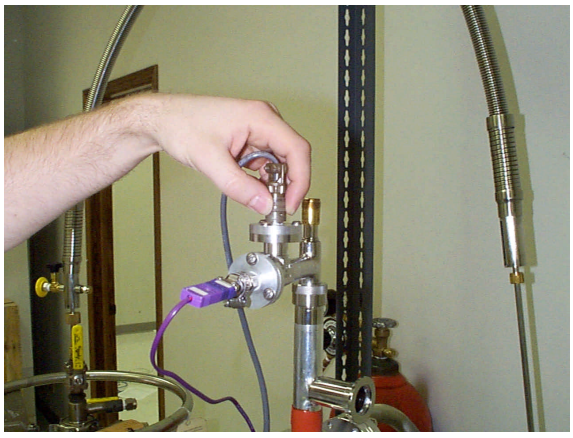

Figure 24

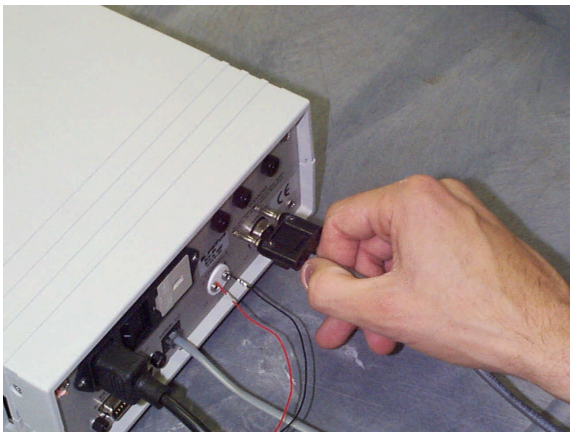

Figure 25

27. Connect the sample heater to the temperature controller with the cable provided (Figure 24 and Figure 25).
28. Connect the temperature controller to the vacuum pumping station Isobar using the cord provided.
29. Connect Active Gauge Controller cable labeled “Cryostat Vacuum” to the gauge on the cryostat vacuum chamber (Figure 26).

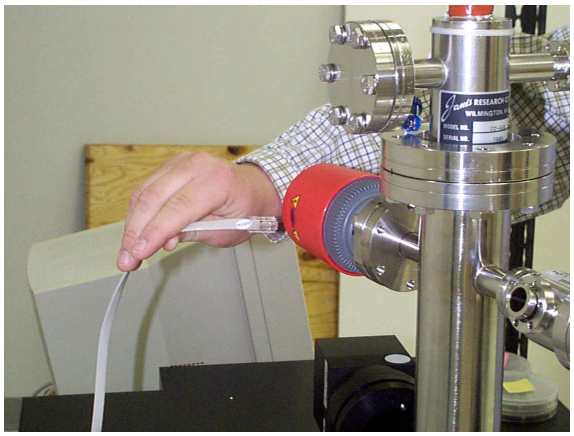

Figure 26

This completes the installation of the CRYO-200 option on the ellipsometer base. The ellipsometer installation may now be completed.

The cryostat was shipped with a thermal oxide (~250Å) on silicon sample mounted on the cold finger. Ellipsometer and data acquisition test can be performed.

Turbo Pump Operation And Seal Checkout

Some general trouble-shooting and operational comments are presented below. UHV systems have a complex nature which is beyond the scope of this manual. For an in depth understanding of UHV, consult relevant books, journals, and experienced colleagues.

- Leaks have a large number of causes. The most obvious are:
    - ‘Recycling’ used copper gaskets. You might get lucky and get one to seal, but how much time do you want to spend fixing a known source of trouble? Always use fresh copper gaskets when assembling a UHV seal.
    - Improper torque sequence. Torquing of the seal bolts in an uneven manner will cause uneven compression of the gasket causing it to leak. Loosen and re-torque seal bolts in the prescribed fashion. If the torquing is really bad, the gasket will have to be replaced.
    - Inadequate torque. Seals rely on bolt tension to compress the gasket and hold it in intimate contact with the seal surface. Torque the seal bolts to the prescribed value.
    - Dirt on seal mating surfaces. Dirt keeps the gasket from coming into intimate contact with the seal surfaces and may damage the seal surfaces. Always check your seal surfaces for foreign material (dirt, lint, dust, hair, etc.) before assemble a UHV seal. Replace the gasket and clean the seal surfaces.

- During pumpdown, there can easily be two (or more) orders of magnitude difference in the pressure between the pump and cryostat chamber. Even after pumpdown, there can be an order of magnitude difference.

It is recommended to keep the vacuum line and cryostat shell under vacuum to prevent water adsorption.

- If the system is well sealed, water vapor will be the major contaminant in the chamber and it can take weeks to pump out. Therefore it is recommended to keep the flexible vacuum line and the cryostat shell under vacuum to prevent water adsorption. For extended idle times, the valves can be closed and the turbo pump turned off.
- The start/stop cycle of the pumping station is an automated sequence, and may not immediately appear to be functioning.

The following procedures will check the integrity of the three copper gasket seals just installed. The seal just above the turbo pump should have already been checked before assembling the table, but check it again at this time.

Check seal between pump and vacuum tee.

1. Make sure the main vacuum valve and the cryostat vacuum valve are completely closed.
2. Connect the pumping station Isobar to AC power (separate circuit from the ellipsometer).
3. Make sure the power switch for each of the pumping station components is in the 'on' position (turbomolecular pump controller, active gauge controller, and roughing pump) as well as the main power switch (all switches are located on the back side of the pumping station).
4. The pumping station should be powered and the displays active. Toggle the vacuum gauge display to read the pump vacuum (see the Edwards Active Gauge Controller manual).
5. Start the vacuum pump by pressing the 'Start' button. The pressure should quickly drop to below 10^{-4} Torr. If not, there is probably a leak in the seal between the turbomolecular pump and the vacuum tee. If there is a leak, try tightening th clamping bolts. If the leak persists, press the 'Stop-Start' button to stop the pump, and let the system return to atmospheric pressure. Replace the copper gasket seal and try pumping down again.

Check seals at ends of the vacuum transfer line.

6. While monitoring the pump vacuum gauge, very slowly open the main vacuum valve such that the pump gauge pressure never exceeds 1 Torr. Once the valve is fully open, the pressure should quickly drop to below 10^{-4} Torr. If not, there is probably a leak in one of the seals at the ends of the flexible vacuum hose. If there is a leak, try tightening clamping bolts. If the leak persists, close the main vacuum valve, but the turbo pump can be left running. Replace the copper gasket seal(s) and try pumping down again.
7. Wait for the pump gauge pressure to reach 10^{-5} Torr.

Check cryostat seals.

8. While monitoring the pump vacuum gauge, very slowly open the cryostat vacuum valve such that the pump gauge pressure never exceeds 1 Torr. The cryostat was shipped under vacuum so the pressure should not rise appreciably, but go slowly for safety. Once the valve is fully open, the pressure should quickly drop to below 10^{-4} Torr. If not, there is probably a leak in one of the seals on the cryostat. If there is a leak, try tightening the clamping bolts. You may want to close the cryostat vacuum valve and confirm that problem is really with the cryostat itself. If the leak persists, start the seal chck out procedure again to isolate which seals could possible be at fault. Replace the copper gasket seal(s) and try pumping down again.

Exchanging Cryostat And Standard Sample Stage

Cryostat To Sample Stage Exchange

Tools Needed:

- 4mm key wrench
- 3/16” key wrench

Parts Needed:

- Sample stage components:
- Sample chuck assembly, 1 each
- Z-axis stage, 1 each
- ¼-20 by 3/8” socket head cap screws, 6 each

Procedures:

1. The cryostat storage pedestal should be positioned behind the ellipsometer, straddling the ellipsometer base connecting cables.
2. Set the system angle of incidence to 0° (via the Move Angle command).

The copper radiation shield does not warm up or cool down as fast as the cold finger.

3. Allow the cold finger to come to room temperature. If the cryostat has been cold, the temperature controller may be set to 295 Kelvin to quickly warm up the sample. If the cryostat has been hot, cryogen may be used to cool the sample.
4. Completely unscrew the transfer line O-ring compression nut and carefully withdraw the transfer line bayonet from the cryostat by pulling the transfer line straight up (Figure 27 and Figure 28). A step stool may be necessary to accomplish this step. Caution: The bayonet may be cold! Observe proper safety precautions!

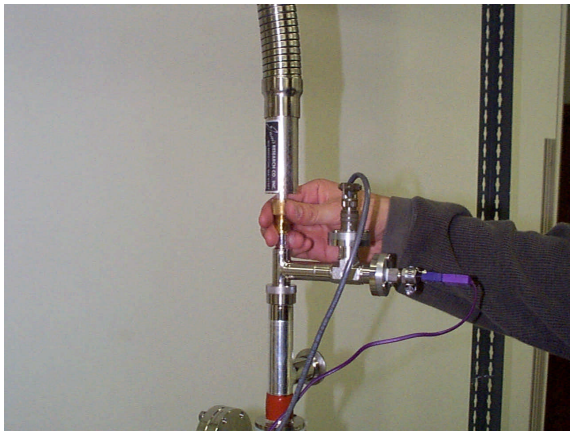

Figure 27

Figure 28

5. Using the 4mm key wrench, remove the four M5 socket head cap screws that hold the cryostat assembly to the goniometer base (Figure 29). These screws will be used to fasten the cryostat assembly to its storage stand.

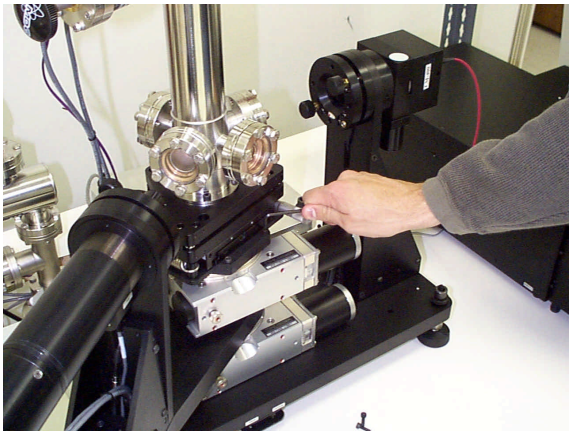

Figure 29

Be Careful! The cryostat and tilt stage are heavy.

6. Using care not to strain the flexible vacuum hose (if attached to the cryostat chamber), gently move the cryostat assembly to its storage stand (Figure 30).

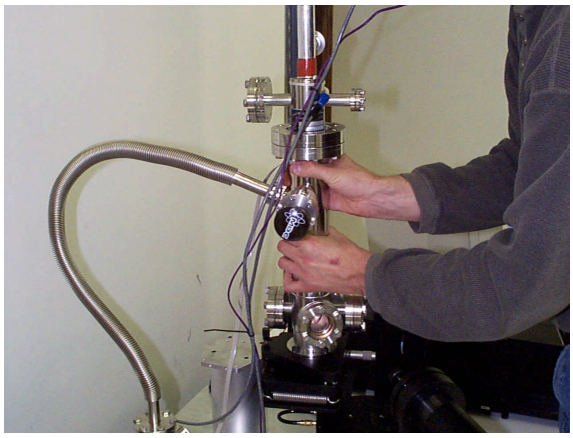

Figure 30

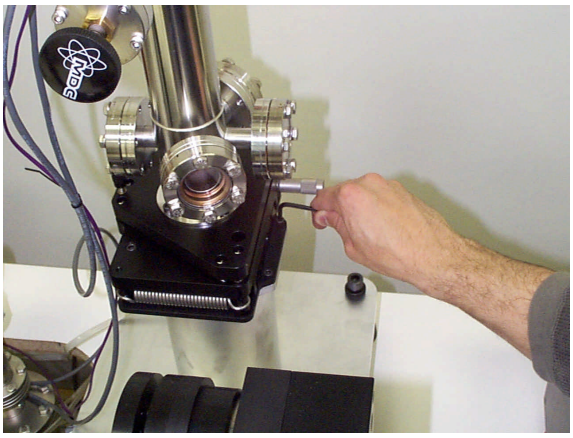

Figure 31

7. Install the four M5 socket head cap screws finger tight through the CRYO-200 base into the storage stand.
8. Using the 4mm key wrench, tighten the four M5 socket head cap screws (Figure 31).
9. Using the 3/16” key wrench, bolt the sample z-axis stage to the goniometer base with two ¼-20 socket head cap screws (Figure 32).

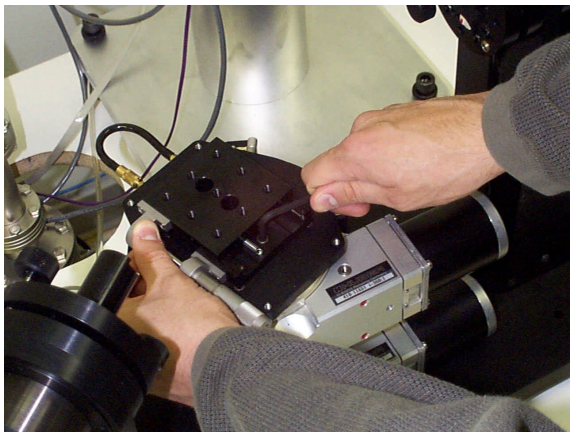

Figure 32

10. Using the 3/16” key wrench, bolt the Sample Chuck on the sample z-axis stage with four ¼-20 socket had cap screws (Figure 33).

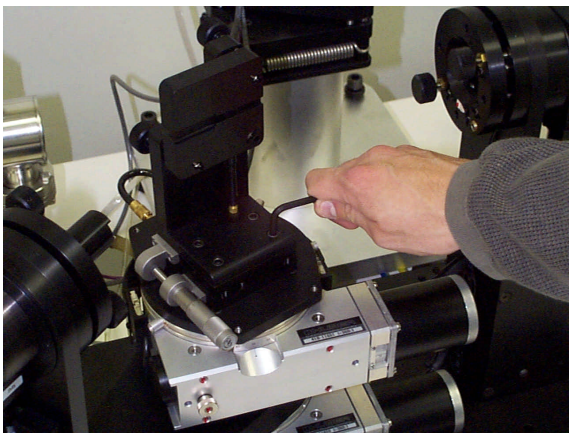

Figure 33

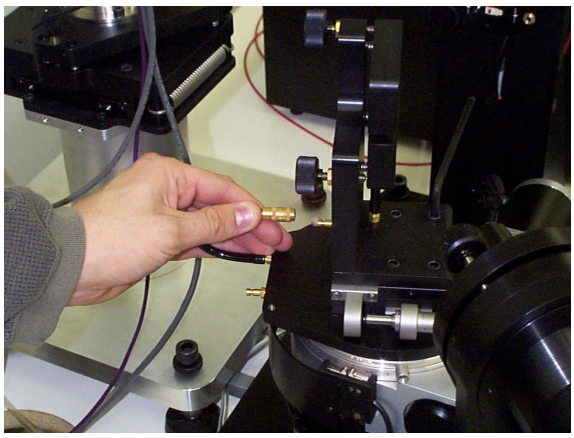

Figure 34

11. Attach the sample vacuum to the sample chuck (Figure 34).

Make sure goniometer dials agree with the WVASE32 settings.

12. Check the angles in the Motor Settings and make sure they agree with the goniometer positions. Zero the fine scales on the goniometers if necessary (See VASE Hardware Manual).

Sample Stage to Cryostat Exchange

Tools Needed:

- 3/16” key wrench
- 4mm key wrench

Procedures:

1. Set the system angle of incidence to 0° (via the Move Angle command).
2. Disconnect the vacuum from the sample chuck (Figure 35). Connect the vacuum hose to the dummy connection on the sample goniometer.

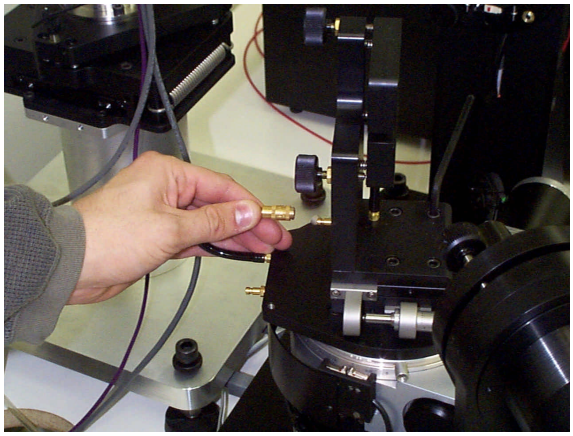

Figure 35

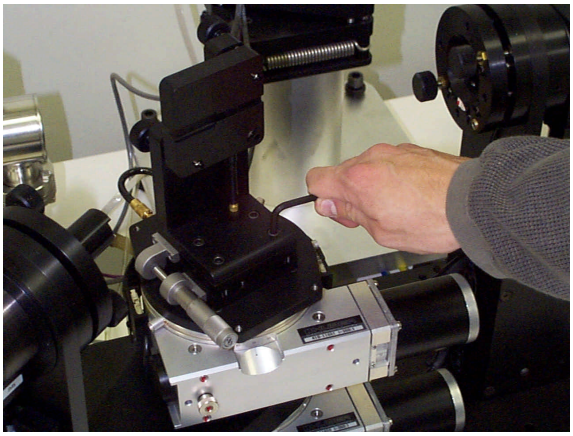

Figure 36

3. Remove the four ¼-20 socket head cap screws that hold the sample chuck to the sample Z-Axis stage (Figure 36).
4. Remove the two ¼-20 socket head cap screws holding the sample z-axis stage to the sample goniometer (Figure 37).

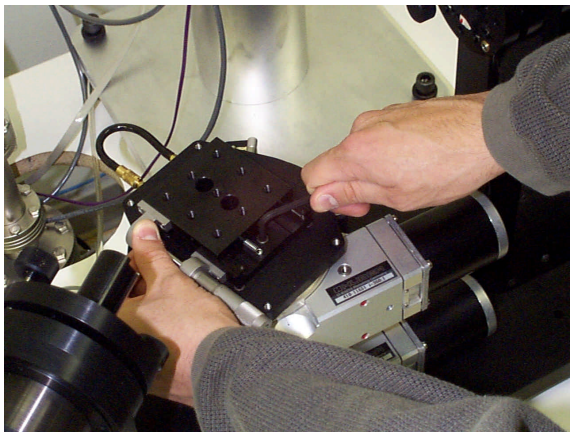

Figure 37

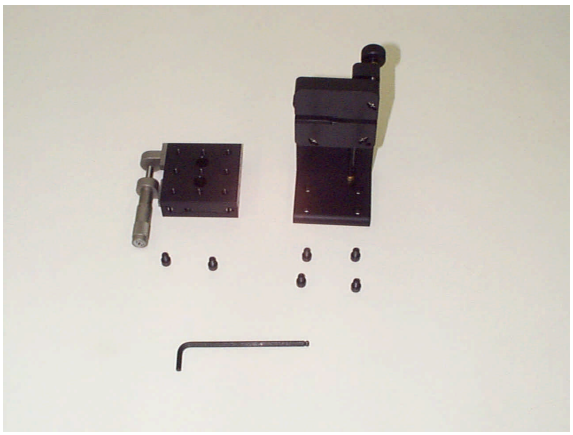

Figure 38

5. Store the removed Sample Stage components for reuse (Figure 38).
6. Using the 4mm key wrench, remove the four M5 socket head cap screws holding the cryostat assembly to its storage stand (Figure 39). These bolts will be used to fasten the cryostat assembly to the goniometer base.

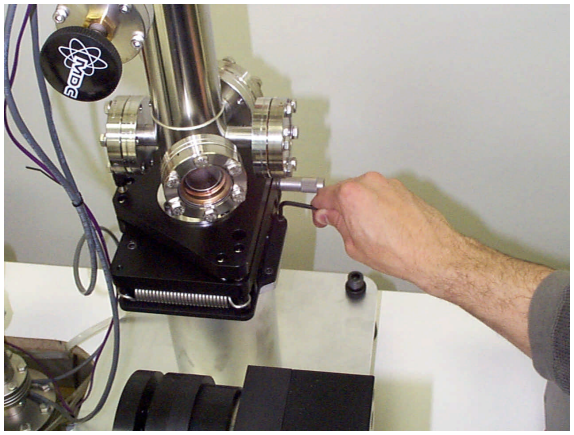

Figure 39

Be Careful! The cryostat and tilt stage are heavy.

1. Using care not to strain the flexible vacuum hose (if attached to the cryostat chamber), gently move the cryostat assembly to the goniometer base with the Z Axis micrometer knob on the input unit side (Figure 40).

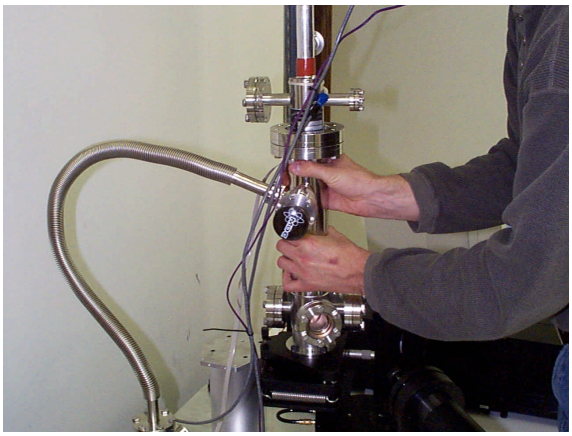

Figure 40

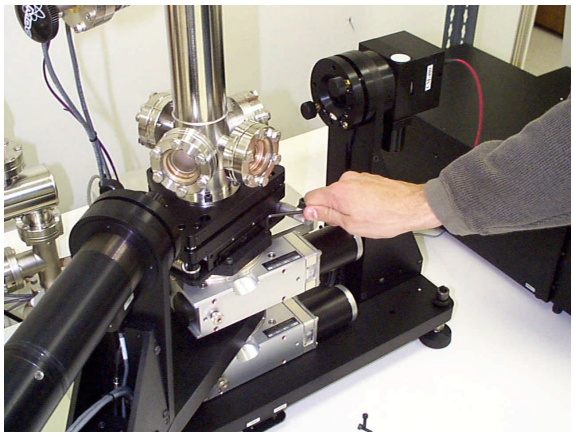

Figure 41

7. Install the four M5 socket head cap screws finger tight through the CRYO-200 base into the ellipsometer base.

8. Using the 4mm key wrench, tighten the four M5 screws (Figure 41).

Note: **Keeping the cryostat vacuum chamber under vacuum during storage is highly recommended.** This removes the possibility of chamber contamination, minimizing the pump-down time required before usage.

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Section 3 Configuring WVASE32® For Cryo-200 Attachment

Window Strain Effects And Window Calibration

Obviously windows are needed on a vacuum chamber to permit an optical probe to reach the sample without letting the vacuum "leak out". However, with windows comes the possibility of strain effects which can perturb the polarization state of the probe beam. These effects and the general WVASE32® scheme for handling them are discussed below.

The Cryo-200 comes with standard, fused-silica, UHV windows, and these windows generally have some strain. Expensive, fragile "strain-free" windows, which might be more appropriately named "lower-strain-when-shipped", have been developed. Regardless of the window type, if there is detectable window strain (the usual situation) then that strain needs to be accounted for, not ignored, and the accuracy of the strain model will be about the same whether a small or moderately-large strain effect is present.

In general terms, the effect of window strain can be divided into two parts. Part 1 is a pure window effects which can be determined when the ellipsometer is calibrated for a very wide range of samples. Part 2 is a window effect which merges completely with the measured Δ value for the sample. Part 1 effects are referred to as WinEffects, and Part 2 effects are referred to as DelOffsets. (See hardware.cnf section below.)

With windows, the measured y and D values must be interpreted as applying to the window-sample-window system as a whole.

One tractable, but unfortunate, consequence of windows combined with a rotating-analyzer ellipsometer is that the window effects can not be fully disentangled from the measured ψ and Δ values at acquisition time. Without windows, a rotating-analyzer system has an ambiguity with respect to sign of Δ which is not a serious problem because Δ can be mapped back to the 0-180° range for analysis. However, with windows present, the measured Δ relates to the combined window-sample-window system and there is no general, convenient mapping to apply. With windows, the measured ψ and Δ values are not intrinsic sample parameters. The measured ψ and Δ values must be interpreted as applying to the complete window-sample-window system. WVASE32® manages this complication by tagging the data files with the appropriate window effects information which is automatically loaded into the Model Options when the data file is loaded.

Data files are tagged with the appropriate window effects information for use when analyzing the data.

To analyze multiple-angle cryostat data, use multiple models, with data at one angle per model.

The spread of strain effects across the window also needs to be considered if data is to be taken at more than one angle. As discussed in the next section, the Cryo-200 attachment has its windows divided (via software) into different zones for different angle of incidence ranges. As data is acquired at different angles, window effects for that angle zone are tagged with the data file. However, for analysis, any single model and corresponding experimental data can have only one set of window effects. Therefore, one can not simultaneously model cryostat data acquire at different angles of incidence using a single model. To analyze multiple-angle cryostat data, use multiple models, with data at one angle per model.

For normal operation, it is not necessary to recalibrate window effects. Previously stored values will be used.

The Part1, WinEffects, values can in principle be determined with each normal hardware calibration. However, the default operating procedure for the Cryo-200 is to not fit new window effects. With the Cryo-200 present the following additional dialog box is displayed during the normal calibration procedure. For normal operation, the recommend action is to select 'No' at this stage.

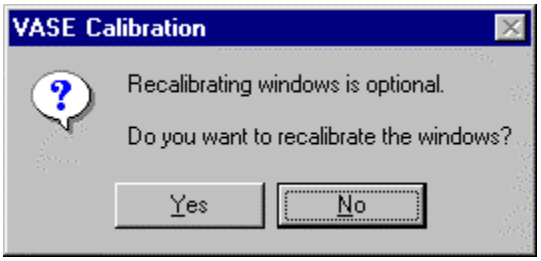

Figure 42

A WinEffects system calibration requires a thermal-oxide calibration sample.

If the WinEffects are to be recalibrated, then it is recommended that a full window system calibration be performed. A window system calibration determines appropriate WinEffects and DelOffsets. A system calibration entails loading a piece of the thermal-oxide calibration wafer (provided with the system) as the cryostat sample and then running a special script file. Because the system calibration is a combined spectroscopic calibration and model fit, the sample need only have an oxide thickness near the nominal 250Å. (Other simple samples, which can be fit to high accuracy without windows present, could also be used.) The data acquisition and analysis takes about two hours. If the windows are removed or substantial external heat is applied to bake out the chamber, the windows will need to be recalibrated. Otherwise, the windows are typically stable.

Procedures:

1. The CRYO-200 was shipped with the correct calibration sample inside. If it is still present you can skip the steps relating to installation of a new sample.

2. Remove the cold finger assembly referring to procedures described in the section on “Removing Cold Finger Assembly From The Cryostat And Changing Samples.”
3. Place the calibration sample on the cold finger.
4. This calibration should be performed near room temperature (~297K) and does not require a vacuum. If this sample is only going to be used to calibrate the windows, then it is probably unnecessary to replace the copper gasket when returning the cold-finger assembly to the cryostat. (If the system is going to be pumped down, then replace the seal.)
5. Perform a proper coarse alignment as described in the section on “Inserting Cold Finger Assembly In To The Cryostat And Aligning The Sample.”
6. The seal clamping bolts should be tightened enough to prevent the sample from moving around. Then a fine alignment should be performed.
7. Perform a fine calibration at 70° and select fitting for window effects when that dialog appears. (Figure 44) If the calibration is successful, continue with the system calibration. If the fine calibration was unsuccessful, you may need to perform a coarse calibration. It is important to confirm that the system is capable of being calibrated before starting the multiple system calibrations that follow.
8. Launch the WSCRIPT.exe script running application using the |Global|Run_WVASE_Tools menu. See *Figure 43*.
9. Load the ‘ftdelwin.wsc’ script file. See *Figure 44* below.
10. Run the script. This should take about two hours.
11. The first time this procedure is completed without a J.A. Woollam Co. representative in attendance, the user should e-mail the company the “\wvase32\current.log” file for validation of the procedure.

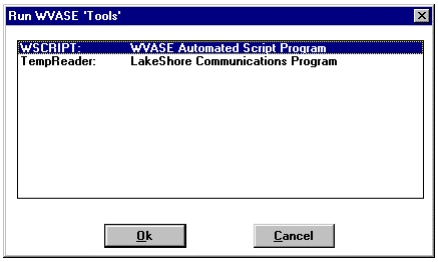

Figure 43

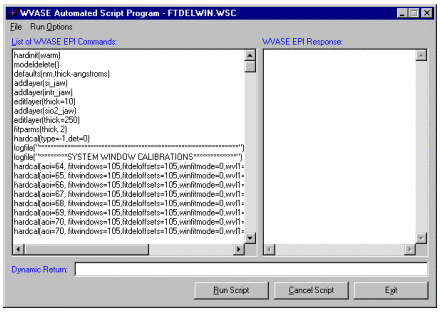

Figure 44

Hardware.cnf Entries For The Cryo-200

The hardware.cnf file contains the hardware configuration information that tells WVASE32® what kind of ellipsometer system is available and how to control that system. Configuration entries specific to the Cryo-200 are described below.

In the [Hardware] section of the hardware.cnf file, the following entry identifies 'attachment1' as 'cryostat'. In turn this causes WVASE32® to look for the [cryostat] subsection for more configuration entries.

```
[Hardware]
attachment1=1 cryostat
```

The following lines provide acquisition guidelines for WVASE32® to use when the presence of the Cryo-200 is detected.

```
[Cryostat]
switchconfig=27 1
AllowAlignJogs=1
fixedpolalways=1 20
;zoneavealways=1
```

The 'switchconfig' entry tells the program that when sensor bit #27 is active, the Cryo-200 is present. There is a proximity switch in the sample stage base that detects the presence of the cryostat. The 'AllowAlignJogs' entry enables the left/right arrow keys to be used for left/right, fine sample alignment, but only with the Cryo-200 present. The 'fixedpolalways' entry tells the program that the polarizer must be fixed for a given scan (needed because of possible window effects), and that the fixed polarizer azimuth must be 20° (or ±20° if zone averaged). The 'zoneavealways' entry, which is commented out (not active) in the above example, would force zone averaged data acquisition. While this is not an absolute requirement, zone averaging the polarizer is highly recommended, even without possible window strain.

The Cryo-200 should be used at integer angles of incidence and is principally designed to work at 70°

The remaining [Cryostat] entries define the angle zones for the windows, define how window effects will be calibrated, and specify the current window effects are. As shipped, the ellipsometry windows were divided into 14 zones, most of which are 1° wide and centered on an integer angle. These values should not be directly modified by the user. Any changes in the current window effect values. will be entered by WVASE329® as the results of a calibration.

```
[Cryostat]
nZones=14
angzone=70 69.5 70.5
angzone2=0 -10 10
angzone3=71 70.5 71.5
angzone4=72 71.5 72.5
angzone5=73 72.5 73.5
angzone6=74 73.5 74.5
angzone7=75 74.5 75.5
angzone8=76 75.5 80
angzone9=69 68.5 69.5
angzone10=68 67.5 68.5
angzone11=67 66.5 67.5
angzone12=66 65.5 66.5
angzone13=65 64.5 65.5
angzone14=64 50 64.5

wineffects=2 -1.34693 -1.50901 0.00178 0.00056
winfitmode=2
```

```
wineffects2=2 0.00000 1.21400 0.05869 -0.00282
winfitmode2=12
wineffects3=2 -1.29704 -1.39076 0.00583 0.00047
wineffects4=2 -1.16826 -1.25907 0.00278 0.00054
wineffects5=2 -0.85683 -1.04749 -0.00052 0.00069
wineffects6=2 -0.80369 -0.71778 -0.00645 0.00119
wineffects7=2 -0.27786 -0.28113 -0.03636 0.00242
wineffects8=2 0.08332 0.26995 -0.04420 0.00278
wineffects9=2 -1.55471 -1.52092 0.00240 0.00055
wineffects10=2 -1.66636 -1.50378 0.00036 0.00069
wineffects11=2 -1.39243 -1.37807 -0.00363 0.00077
wineffects12=2 -1.30172 -1.21444 -0.00380 0.00066
wineffects13=2 -1.26951 -0.88734 -0.00646 0.00070
wineffects14=2 -1.43652 -0.40560 -0.00191 0.00041

DelOffsets=2 0.45295 0.00178 0.00056 0.500
DelOffsets=2 0 0 0 .5
deloffsets2=2 0 0 0
deloffsets3=2 -0.08044 0.00583 0.00047 0.500
deloffsets4=2 -0.70477 0.00278 0.00054 0.500
deloffsets5=2 -1.27227 -0.00052 0.00069 0.500
deloffsets6=2 -1.58812 -0.00645 0.00119 0.500
deloffsets7=2 -2.30921 -0.03636 0.00242 0.500
deloffsets8=2 -2.85260 -0.04420 0.00278 0.500
deloffsets9=2 1.05519 0.00240 0.00055 0.500
deloffsets10=2 1.80334 0.00036 0.00069 0.500
deloffsets11=2 2.58509 -0.00363 0.00077 0.500
deloffsets12=2 3.45108 -0.00380 0.00066 0.500
deloffsets13=2 4.23914 -0.00646 0.00070 0.500
deloffsets14=2 5.16073 -0.00191 0.00041 0.500
```

The 'User1' value is the temperature passed to WVASE32® by TempReader.exe

The [User] subsection tells the program to look for an external 'User1' value to be used when acquiring dynamic data. For the Cryo-200, the 'User1' value is the temperature passed to WVASE32® by the TempReader.exe utility program.

```
[User]
User1=1 0
User1Cal=0
```

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Section 4 Cryostat System Operation

LakeShore® Temperature Controller, Model 330-5X

The LakeShore manual is the primary reference for the temperature controller.

The primary documentation for the LakeShore® Model 330-5X Temperature Controller is the LakeShore manual shipped with the system. Only some of the important features and operational considerations are discussed here.

The extended Type-E thermocouple used from 4-700K is not standard*. See the LakeShore manual addendum and the TempReader.exe description.*

The Cryo-200 is equipped with an extended Type-E thermocouple to monitor temperatures from 4 to 700K. The LakeShore controller does not have a calibration curve for this extended range thermocouple stored in permanent memory (curves 1-10, See LakeShore page 2-8). The controller is shipped with extended Type-E calibration values stored Curve #12 in non-volatile RAM. The curve will remain stored in memory during normal operations including turning the power off, but the curve can be deleted by resetting to factory defaults (See LakeShore page 3-13). If the curve becomes lost or corrupted, a new curve must be downloaded before correct temperature measurements are possible. There is a special format for downloading an extended Type-E calibration which is found in the one page addendum to LakeShore manual. The TempReader.exe utility program can be used to download new calibration curves as discussed in the next section.

It the temperature controller oscillates badly, examine the "P" (proportional) control parameter. It may need to be manually adjusted.

On the Cryo-200, the LakeShore controller regulates temperature solely by adjusting power delivered to a resistive heater embedded in the cold finger. Thus, to maintain temperature control, heat dissipation either by evaporating cryogen (below room temperature) or by conductive/convective losses (above room temperature) is required to pull against the heater. Because the pull-down heat dissipating forces in the system (e.g. cryogen flow rate) tend to be highly variable, it is difficult to determine universally optimal PID control parameters. The LakeShore controller is capable of sophisticated multi-zone temperature PID control functions which are outside the coverage of this manual, however, the user is encouraged to try using the AutoTune feature of the controller for most control applications. Occasionally the AutoTune feature will drive one of the PID control parameters too far in one direction requiring the user to make a manual adjustment (see LakeShore page 3-10). For instance, if temperature control oscillates very badly, the P (proportional) value may be too large, e.g. 650, and needs to be reset to a more stable value like 20.

There are several factors which may affect accuracy and precision of the monitored thermocouple temperature. The users should be aware of these potential limitation and should gauge their importance relative to the particular experiment under consideration.

1) Thermocouple voltage measurements may have some offset induced by undesired junctions in the wiring. These are effects are reduced by the cold junction compensating device in-line with the thermocouple. Additional offset correction capability is available using the TempReader.exe utility to down load a modified calibration curve to exactly match one or two known temperatures to a measured voltage.

2) The extended Type-E thermocouple which covers a very wide temperature range, is much less sensitive to temperature changes around 4K than it is around 300K.

3) The sample temperature may not be exactly the same as the thermocouple. The thermocouple is in close proximity with the heater and helium outlet in the cold-finger assembly. The thermocouple needs to be close to the hot/cold source for control stability (see "Introduction to Laboratory Cryogenics" page 65). However, this permits a temperature offset to exist between the sample and the thermocouple. Furthermore, the offset between sample and thermocouple may depend not only on what the control temperature is, but also on how much cooling/heating is taking place to maintain that control temperature. An experiment using a secondary temperature probe may be possible to characterize the sample-thermocouple offset as a function of control temperature and controlling heater power.

4) There is a time lag between the sample temperature and the thermocouple monitor temperature for the same reasons discussed in point 3 above.

TempReader.exe Utility Program

Make sure the LakeShore baud rate is set to 1200. See LakeShore page 3-13.

The TempReader utility written by J.A. Woollam Co., is a simple program that performs just the three basic tasks as described below. It is not a general control program for the LakeShore. If a more sophisticated WVASE32®-LakeShore® interfacing program is required for a particular experiment, the user will need to contact the J.A. Woollam Co., for more information on the WVASE32® external programming interface (EPI). A screen shot of the inactive TempReader window is shown below. TempReader can be started from the Global|Run_WVASE_Tools menu from inside WVASE32®, or it can be launched directly from the operating system out of the \wvase32\tools subdirectory. TempReader assumes a baud rate of 1200 bit/s using the computer's ComPort #1. If these values need to be changed, contact J.A. Woollam Co. for more information.

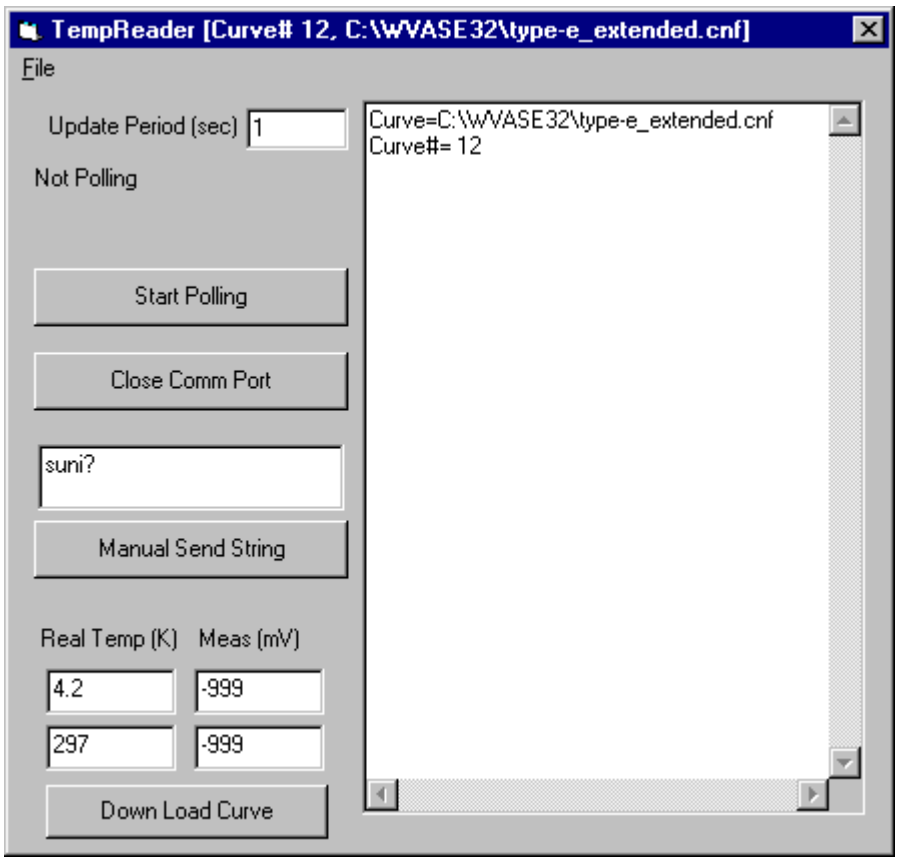

Figure 45 TempReader.exe front screen.

1) TempReader can poll the current sensor temperature and pass that value along to WVASE32®.

The utility can be used to poll the current sensor temperature from the controller using a serial communications port, and then advise WVASE32® what the current temperature is. WVASE32® can then store the current temperature (USER1 value) with the measured ellipsometric data. (See Acquisition and Analysis Section) The polling controls are in the upper left hand corner. The update period can be selected between 0.5 and 60 seconds. The same button is used to Start Polling and Stop Polling. The Close Comm Port button may help clear the program if the program gets caught in a series of repeated errors. Error information is presented in the text box on the right side. Serial communications under Windows® tends to produce some errors. As a general matter, TempReader deals with these errors by ignoring them and then restarting the polling process. The goal is to send only correct temperatures to WVASE32®, thus not all polling attempts are successful.

2) TempReader can download a thermocouple calibration curve and apply an optional user-defined correction.

TempReader can also download a thermocouple calibration curve. By default, TempReader works with a file called "type-e_extended.cnf" and curve #12. The currently configured curve# and file are displayed in the TempReader text box when the application is launched. (See Figure 45.) The default curve# and filename can be changed by using command-line arguments which are accessible using a Windows® shortcut to launch TempReader. For example, if the command line contains "curve#=13, curve=type-e_alternate.cnf", then TempReader will look for a file called "type-e_alternate.cnf" and download the values to curve #13 if directed to do so. The format for a thermocouple ".cnf" file can be seen the in the text of "type--e_extended.cnf" at the end of this section.

A linear, user-specified correction can be applied to the calibration curve before downloading using the 'Real Temp' and 'Meas' text boxes in the lower left corner. By using known cryogenic control temperatures, such as imersion in liq. helium or liq. nitrogren, the actual measured thermocouple voltage can be measured by selecting the mV units display. (See LakeShore 3-3) A measurement with the cold finger assembly outside the cryostat and at equilibrium at room temperature can provide another known temperature, assuming a secondary accurate temperature measurement is available. It is not recommended to directly immerse the cold finger in ice water because water vapor is primary background gas at the vacuum pressures typical for the cryostat. In all cases, remember that even if the thermocouple were perfectly calibrated, the temperature of interest is the temperature of the sample. (See end of previous LakeShore® Temperature Controller section.)

In Figure 45, the 'Meas' boxes contain -999 which cause NO correction to be applied. If exactly one of the 'Meas' boxes contains a value which is not -999, then TempReader will adjust the downloaded calibration voltages by a constant offset to match the actual measured value for the specified temperature. Of course the preferred method is to make the measured voltage match the thermocouple values using proper wiring techniques and the ice-point buffer module. If both 'Meas' boxes contain values which are not -999, then a linear correction to the downloaded voltages is made such that the measured values match both specified temperatures. A two-point correction effectively shifts and tilts the calibration curve to match the known measured points.

TempReader does NOT perform a plausibility check on the user defined correction.

Note, TempReader does not perform a plausibility check on the user defined correction. Therefore, very inaccurate temperature read outs can occur. A potential hazard exists if the LakeShore is instructed to control the temperature with an invalid calibration curve. For example, the heater might turn on and remain at full power indefinitely because the desired temperature can never be reached. (The same thing would also happen if an unreachable temperature was selected as the control point, even with accurate calibration values.)

3) TempReader can pass commands to the controller to test the serial link and to examine the controller NOVRAM.

TempReader can also pass single commands to the LakeShore and read the returned string. The Manual Send String button sends the text from the box above the button and waits for a return if the string contained a '?' characters. (See LakeShore 4-8 to 4-22 for a complete description of commands). Do not send commands to the LakeShore while polling is active.

The primary purpose of this feature is to test the serial link to the controller. The sensor units query command 'suni?' shown in Figure 45 will return a 'K', 'C', or 'mV' depending on the display of the controller. If this command succeeds, one is assured that serial communication has been established.

There are a variety of commands used to control and query the LakeShore controller. Tests with this feature may be a useful way for the user to develop a separate, more sophisticated control package. Also, some of the NOVRAM values are more easily interrogated using the serial link. Text can be clipped from the log box and pasted into different applications for printing.

File "type-e_extended.cnf" contains the following text. The tabulated values are measured voltage in mV and corresponding temperature in degrees Kelvin. Please note, that for an extended-type thermocouple, both columns of numbers are modified before downloading to the LakeShore controller. See the LakeShore Model 330-5X Addendum for more information.

```
S99EXTENDED TYPE E
P
-15       0
-9.835    3
-9.831    5
-9.821    8
-9.813    10
-9.797    13
-9.777    16
-9.746    20
-9.642    30
-9.503    40
-9.332    50
-9.129    60
-8.899    70
-8.642    80
-8.362    90
-7.733    110
-7.02     130
-6.231    150
-4.916    180
-3.459    210
-1.874    240
-0.176    270
1.617     300
4.786     350
8.168     400
11.73     450
15.435    500
21.189    575
27.106    650
33.122    725
39.184    800
43.228    850
```

Removing Cold Finger Assembly From The Cryostat And Changing Samples

Note: Wear insulated gloves while handling cold or potentially cold items. Wear latex or suitable clean room type gloves while handling parts internal to the cryostat to avoid contaminating them.

Tools Needed:

- Insulated Gloves, 1 pair
- ½" combination wrench, 1 each
- Clean room gloves, 1 pair
- Small straight blade screwdriver
- 3/32" key wrench

Procedures:

1. Set the ellipsometer to the alignment mode and follow the instruction prompts given by the computer.
2. Jog the sample stage angle (fine scale) to zero using the right and left arrow keys on the computer keyboard.

Do not vent the cryostat vacuum shell until the sample and raditation shield have come to room temperature!

3. Allow the cold finger to come to room temperature. If the cryostat has been cold, the temperature controller may be set to 295 Kelvin to quickly warm up the sample. If the cryostat has been hot, cryogen may be used to cool the sample. **Note:** Do not vent the cryostat vacuum shell until the sample and raditation shield have come to room temperature! Parts at elevated temperatures will corrode (permanent) or at reduced temperatures will frost instantly (water contamination is time consuming to remove).
4. Completely unscrew the transfer line o-ring compression nut and carefully withdraw the transfer line bayonet from the cryostat by pulling the transfer line straight up (Figure 47 and Figure 47). **Caution:** the bayonet may still be cold! Observe proper safety precautions.

Figure 46

Figure 47

5. Disconnect the cables from the sample heater, sample thermocouple, and the exhaust port heater.

6. Note: In order to reduce vacuum chamber contamination (which will significantly increase the pump down time), minimize the time the vacuum chamber is exposed to the atmosphere. Be organized, work quickly, do not leave the system lay open unnecessarily.
7. Close the cryostat vacuum chamber valve (Figure 48).
8. Using the ½" combination wrench, remove the eight 5/16-24 cap head bolts that seal the cryostat vacuum chamber (Figure 49).

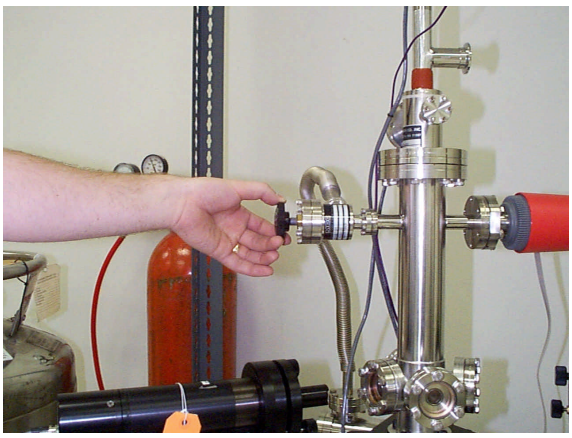

Figure 48

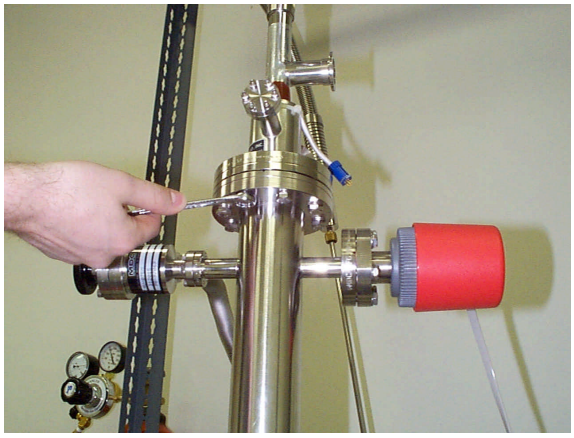

Figure 49

9. Withdraw the cryostat by lifting it straight up out of the vacuum jacket (Figure 50). **Caution:** The radiation shield may still be cold (or hot). Observe proper safety precautions.

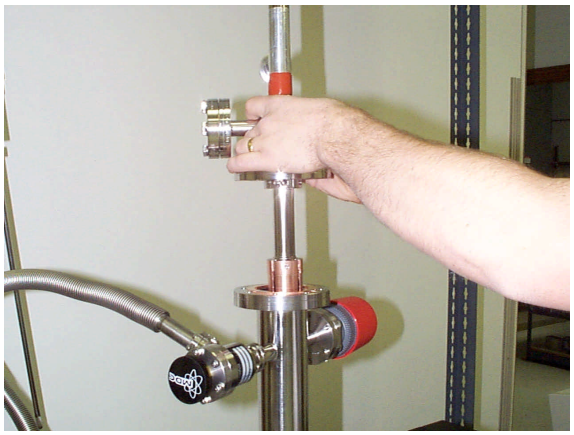

Figure 50

10. Lay the cryostat in its cradle with the sample facing up (Figure 51).

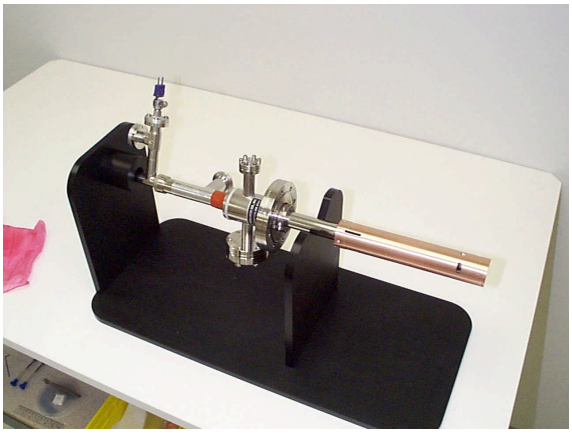

Figure 51

11. Allow the radiation shield to come to room temperature.
12. Using a small straight blade screwdriver, remove the four flat head screws holding the radiation shield to the thermal anchor (Figure 52).

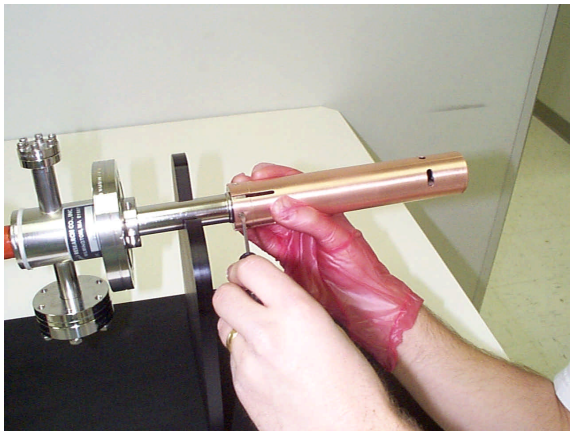

Figure 52

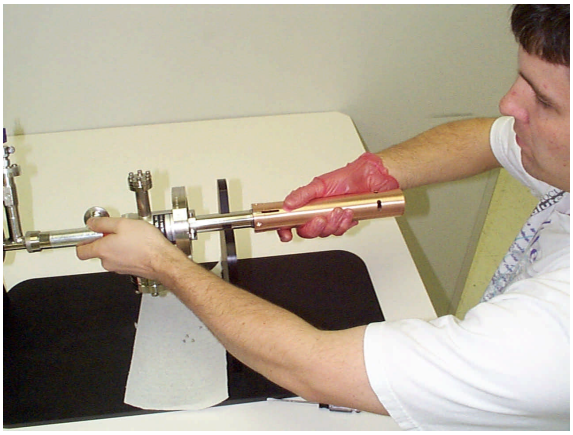

Figure 53

13. Carefully slide the radiation shield off the cold finger (Figure 53).
14. Using the 3/32” key wrench, remove the four screws holding the sample mounting clamps to the cold finger (Figure 54).

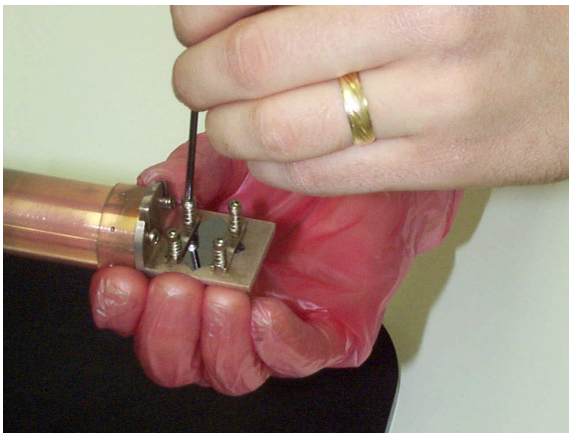

Figure 54

15. Remove the sample mounting clamps.
16. If necessary, adjust the sample chuck height to compensate for sample thickness. This is done via loosening the three screws that mount the sample chuck to the end of the cold finger, sliding the sample chuck such that the face of the sample will be at the center of the cold finger, and securely tightening the three mount screws.
17. Lay sample on the sample mount face up. **Note:** The back surface of the sample has to be flat and clean in order to make good thermal contact with the sample mount.
18. Lay the sample mounting clamps back over their screw holes and insert the hold down screws. **Note:** The mounting clamps should lie across the cold finger, not along the cold finger. (See *Figure 54*.) This will avoid blocking the measurement beam with the clamps.
19. Tighten the screws just enough to hold the sample securely. **Note:** Use the springs provided to help keep from straining (or breaking) the sample.
20. Inspect the radiation shield for frost or condensation. If there is frost, let the radiation shield warm up to room temperature. Wipe condensation off with a clean, soft cloth.
21. Reattach the radiation shield to the thermal anchor with the round view ports (holes) at 0° and 180° and the slotted view ports at 70° and –70°.

Inserting Cold Finger Assembly Into The Cryostat And Aligning The Sample

Tools Needed:

- Insulated Gloves, 1 pair
- ½" combination wrench, 1 each
- Clean room gloves, 1 pair
- Small straight blade screwdriver
- 3/32" key wrench

Procedures:

1. WVASE32® should be running with the hardware initialized and monochromator lamp turned on.
2. Use the |Hardware|Acquire|Align_Sample menu item to start the ellipsometer alignment. There should be a "white light" ellipsometer probe beam and the Hardware window should show an alignment cross-hair.
3. Use the left and right arrow keys in conjuction with optional accelerating modifier keys (shift, cntrl, cntrl-shift) to zero the sample stage goniometer. There is only one move per screen update. If the detector stage goniometer is not zeroed, use the Hardware|Setup|Motors dialog box zero that stage.
4. Using gloves to prevent contamination, remove and dispose of the used copper seal gasket.

5. Inspect the seal mating surfaces on the vacuum jacket and cold finger, clean if any dirt or lint is found to avoid vacuum leaks.
6. Install a NEW copper gasket (Figure 55). Resist the temptation to re-use gaskets, it is an exercise in futility.

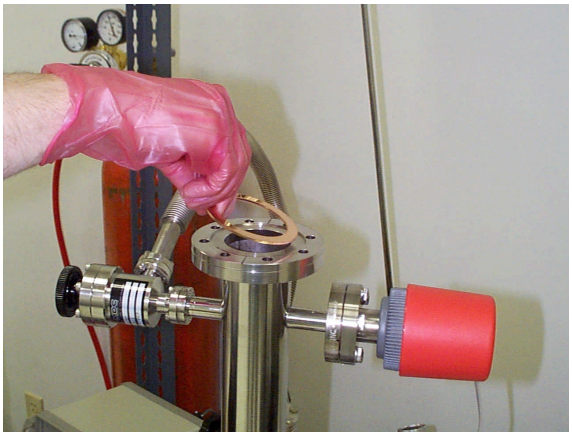

Figure 55

7. Slowly insert the cold-finger assembly into the cryostat shell with the sample surface facing and roughly normal to the white light ellipsometer alignment beam. Try to center the cold finger assembly such that the gasket will be contacted near the final mounting position. Moving the cold finger assembly across the copper gasket may scratch the gasket.
8. Install the eight vacuum seal bolts back into their holes (Figure 56), but do not tighten them (not even finger tight).
9. If necessary, gently rotate the cold finger assembly in the shell to retroreflect the ellipsometer alignment beam back into the quad-detector. The quad-detector intensity increase greatly when nearing alignment. Do not try for perfect alignment at this time, just get the cross-hair on the screen with an intensity much above the background level. For large misalignments, dimming the room lights and using a piece of paper to detect the reflected beam may be useful in reflecting the light back to the quad-detector.

Figure 56

10. Finger tighten the vacuum seal bolts, making sure the sealing flanges are parallel.

Important: Do not over tighten the first bolts. The goal is to produce an even pressure seal all around.

11. Using the ½" combination wrench, gradually tighten the vacuum seal bolts (Figure 57) to 15 lb-ft. Try not to tighten any one bolt more than 10-15 degrees of turn at one time. The recommended tightening procedure is to cycle around the bolts working on every third (skip 2, tighten 1, repeat). The seal flanges should remain parallel throughout the entire bolt tightening sequence.

Figure 57

12. The pump down can then begin.
13. If the vacuum pump is currently off and has been brought up to atmospheric pressure, then open both system vacuum valves and let the cryostat chamber rough out through the turbo pump when the pump is turned on.
14. If the vacuum pump is on but the cryostat chamber is at atmospheric pressure, then the cryostat chamber will need to be slowly released through the turbo pump. Monitor the turbo pump vacuum gauge while very slowly cracking the cryostat gate valve. Try to keep the turbo pump pressure below 1 Torr. About every thirty seconds, or when the pressure is back below 0.1 Torr, open the gate valve a little more, but keep the pressure below 1 Torr. When the pressure no longer increases as the gate valve is opened a little more, proceed to fully open the gate valve. Because the cryostat has a small volume, the cryostat will be mostly evacuated after a few minutes of the above procedure.
15. Inspect the transfer line bayonet for frost or condensation. If there is frost, let the bayonet warm up to room temperature. Wipe condensation off with a clean, soft cloth.
16. Insert the transfer line bayonet into the cryostat (Figure 58)
17. Lightly tighten the transfer line o-ring compression nut (Figure 59).

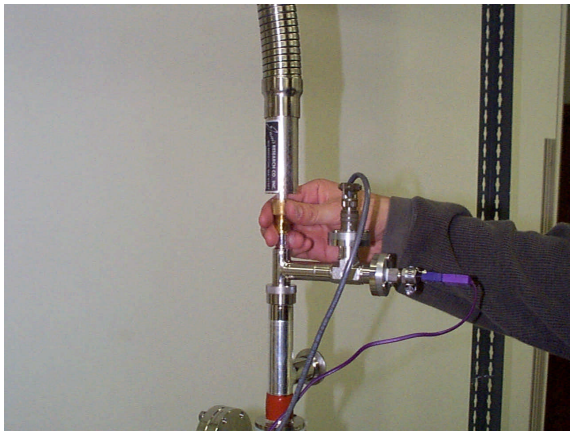

Figure 58

Figure 59

18. With the transfer line installed, the final fine alignment of the sample can be completed.
19. To adjust the Y Axis alignment, use the tilt stage adjustment knob behind the cryostat (Figure 60).
20. To adjust the X Axis alignment, use the left and right arrow keys on the computer keyboard (Figure 61). The sample stage will jog clockwise or counterclockwise one step every time the left arrow or right arrow key is depressed (CTRL-arrow makes 10 steps, and CTRL-SHFT-arrow makes 100 steps).

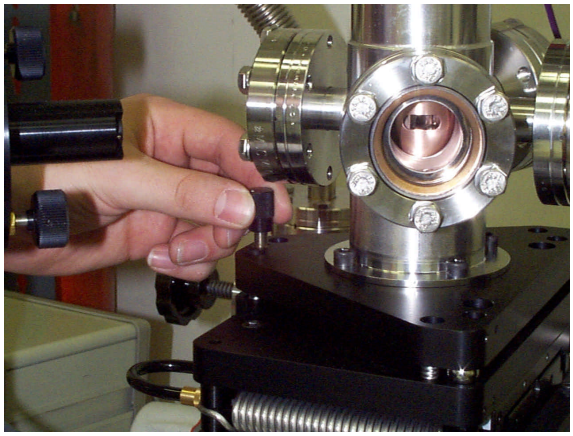

Figure 60

Figure 61

21. When the sample is aligned, press the escape key. Follow the instruction prompts given by the computer.
22. To adjust the Z Axis of the sample, use the micrometer knob on the front of the cryostat tilt stage (Figure 62).

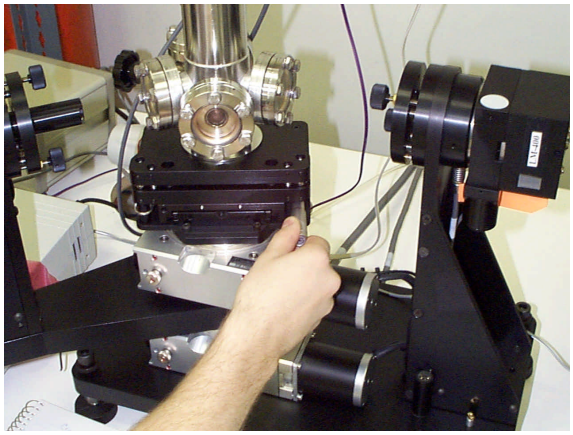

Figure 62

23. Before using cryogen, let the cryostat pump down to below 10^{-6} Torr.
24. Assuming no leaks, the amount of time it takes to pump down is related to the quantity of contaminants being released. If proper UHV handling procedures were observed when changing the sample, the dominant contaminant will be water vapor. In general the amount of water vapor adsorbed on the cold finger and on the interior of the cryostat shell, is directly related to the length of time those surfaces are exposed to atmosphere. If the parts have been exposed to room air for several days, it may require pumping overnight to achieve the desired pressure. If the parts have been exposed for only the several minutes needed to change the sample, pump down may take less than 30 minutes.
25. If you suspect a leak, try tightening the copper gasket clamping bolts in the manner previously described in the sealing section. Sometimes, there is a single bolt which can be tightened to cause a dramatic decrease in cryostat pressure. If the main seal bolts are all sufficiently tightened, then the copper gasket may have a defect. If the gasket is defective it will need to be replaced.

Cryogen Transfer

BE CAREFUL!

Step 1: BE CAREFUL! Cryogens and anything that have been in contact with cryogen are dangerously cold.

The exhaust port heater tape should be turned on before flowing croyogen

While step 1 is universally applicable, the exact procedures to use in transferring cryogen will need to be worked out for the particular setup of the Dewar and choice of cryogen. There is no absolute procedure for initiating and maintaining cryogen transfer. A few operational suggestions are presented below. For additional cryostat operating instructions, refer to “Operating Instructions for the Janis Research SuperTran System” from Janis Research.

Before using cryogen to cool the cold-finger, let the cryostat pump down to 10^{-6} Torr.

Before using cryogen to cool the cold-finger, let the cryostat pump down to 10^{-6} Torr. Once the cold finger assembly becomes cold most of the residual gas in the cryostat will stick to the cold surfaces, including the cold surface of the sample. If you start with less residual gas, there will be less to stick to the sample, and the other surfaces will cryo-pump more effectively if they are not saturated with adsorbed gas during the initial cool down.

There will be variability in the time and manner that the storage Dewar builds up an over pressure and this will in turn affect the rate of cryogen transfer. This variability will be between storage Dewars. Even the same Dewar will change its characteristics as the cryogen fill level changes.

A cold bayonet will ice almost instantly in room air and obviously this ice will not melt away while immersed in liquid helium.

The transfer line bayonet should be warm and dry when inserted into the storage Dewar. Admittedly, this will boil off more cryogen than if the bayonet was pre-cooled with liquid nitrogren for example. However, a cold bayonet will ice almost instantly in room air, this ice can clog the transfer line, and obviously this ice will not melt away while immersed in liquid helium. A wet bayonet is virtually guaranteed to ice closed when inserted into cryogen. In some situations, it may be useful to use the over-pressure created by inserting the bayonet to initiate cryogen flow.

Be patient, it may take awhile to cool the inside of the transfer line.

After opening the bayonet valve on the transfer line (two full turns to start is typical), there may be a substantial wait before liquid cryogen begins to flow. Be patient, it may take awhile to cool the inside of the transfer line. However, if more than twenty minutes have elapsed, then the possibility of icing should be considered. To clear an iced transfer line, remove the bayonet from the cryogen and raise the transfer line to room temperature and make sure all water is off the end of the bayonet before reinsertion.

If the outside of the transfer line between the Dewar and the cryostat should start to frost, then one can probably assume that the vacuum jacket on the transfer line has leaked. In this case, the transfer line will need to be removed, warmed and dried, and the vacuum jacket pumped back down before use. If the transfer line jacket will not hold a vacuum, the transfer line should not be used.

Try to prevent frost formation on the cryogen outflow port.

Proper precautions should be taken keep water and frost out of the ellipsometer.

Once cryogen transfer is proceeding, the transfer rate should be throttled back using the bayonet valve (one full turn open is typical). If an excessive amount of cryogen flows, the heater tape on the cryogen outflow port will be unable to prevent the port from frosting over. When this frost melts it will create water which could then drip into the mechanical and electrical components below. Take precautions to keep this frost (and the water when the frost melts) off of the ellipsometer. Paper towels and styrofoam cups work well for this task.

To hold at the desired operating temperature, the bayonet valve typically only needs to be ¼ - ½ turn open. If the desired temperature is between room temperature and the boiling point of the cryogen, the temperature controller will need to be used. Adjust the flow rate to keep the required compensating heater power in the center of the control range. This way, if cryogen flow rate changes, the controller will be able to adjust power up or down as needed. For temperature controller operating instructions, refer to chapter 3 of “User’s Manual Model 330 Autotuning Temperature Controller” from Lakeshore.

Cycling the sample temperature can reduce the adsorption rate. Details are in the data analysis section “Monitoring Sample Cool Down With Temperature Cycling”

When making low temperature measurements, residual gas in the cryostat will continually deposit on the sample surface. This deposition can be slowed, but not eliminated. In general, a better vacuum (room temperature base pressure) will yield slower deposition rates. Experiments have determined that cycling the sample temperature helps to reduce the adsorption rate. Details of the temperature-cycling procedure are given in the data analysis section “Monitoring Sample Cool Down With Temperature Cycling”.

Section 5 Ellipsometric Acquisition and Analysis

VASE-Dynamic Ellipsometric Acquisition

Most data acquisition runs involving the cryostat will be made using the VASE-dynamic measurement mode.

Most data acquisition runs involving the cryostat will be made using the VASE-dynamic measurement mode. The VASE-Dynamic mode records the acquisition time for each measurement (needed for adsorption-rate analysis at low temperatures) and allows the User1 values (temperatures) to be recorded.

Dynamic data acquisition is started using the Hardware|Acquire|Dynamic_Scan menu option. This brings up the following dialog box.

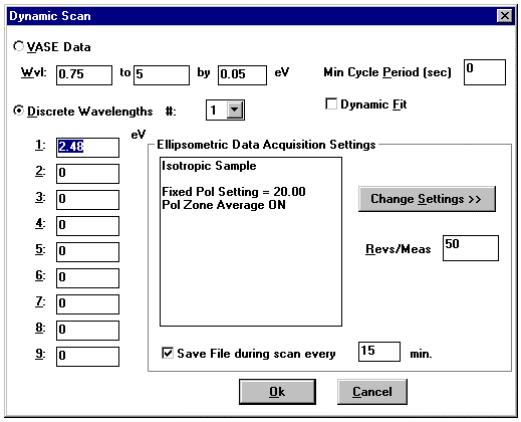

Figure 63. Hardware|Acquire|Dynamic_Scan

Each data point is assigned a measurement time and a cycle grouping time.

Set the angle before starting a dynamic scan.

If **'Discrete Wavelengths'** is selected, then a predefined set of up to 9 wavelengths will be repeatedly measured. Typically, only one or two wavelengths would be used in this case to monitor temperature and/or surface changes, for example. **'VASE Data'** should be selected for more detailed spectroscopic scans. In either case, each data point is assigned a measurement time and a grouping time. The grouping time is just the measurement time of the first wavelength in a spectroscopic cycle. For a single wavelength scan, the grouping and measurement times are the same. The ellipsometric data is acquired using the current angle of incidence.

Additional acquisition settings can be accessed using the **'Change Settings >>'** button which brings up the following dialog box.

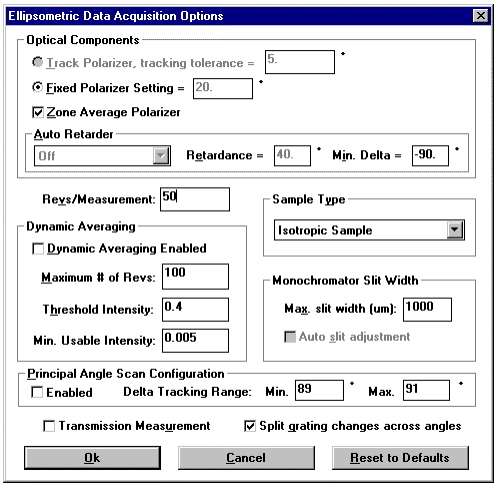

Figure 64

Zone averaging the polarizer is strongly recommended.

In the above example, the **'Zone Average Polarizer'** option has been activated. Zone averaging the polarizer is strongly recommended as the standard measurement mode unless the fastest possible measurement rate is needed.

With the **'Save File During Scan'** option active, the data will be saved to a prenamed file periodically during acquisition. The file name and data comment are entered using the dialog boxes shown below.

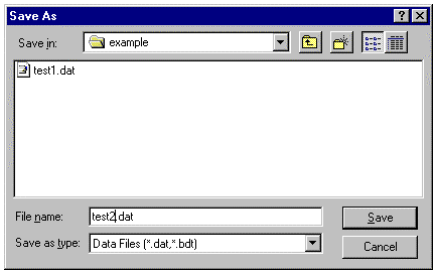

Figure 65

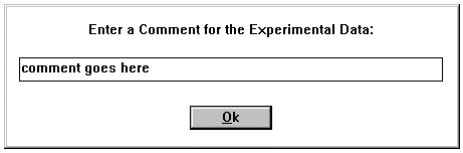

Figure 66

Sample data acquired near room temperature for a heavily sulfur doped InP sample is shown in *Figure 67-Figure 68*. Data from 6 different wavelength cycles are shown in the different Figures. Note, as discussed in "Guide to Using WVASE32", the pseudo-dielectric values $\langle\varepsilon_1\rangle$ and $\langle\varepsilon_2\rangle$ in Figure 68 are just numerically transformed from the ψ and Δ values.

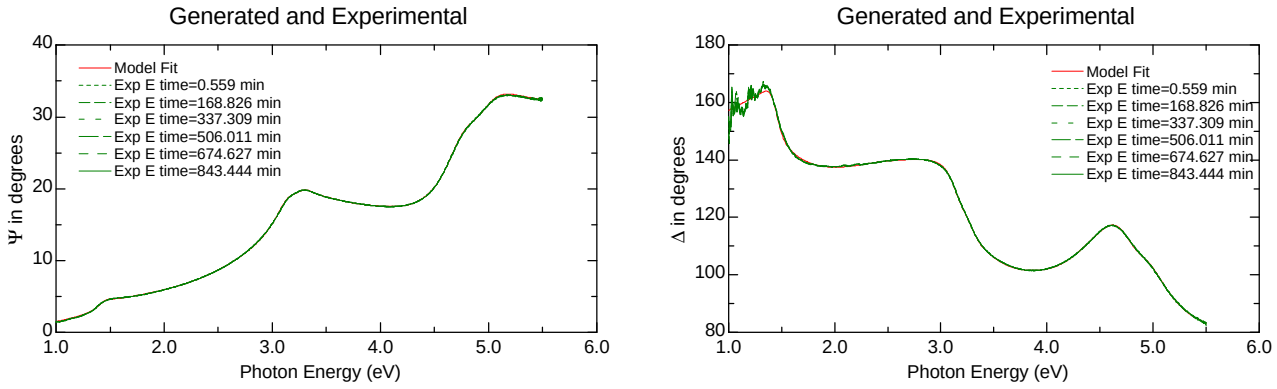

Figure 67a-b. Sulfur doped InP near 297K.

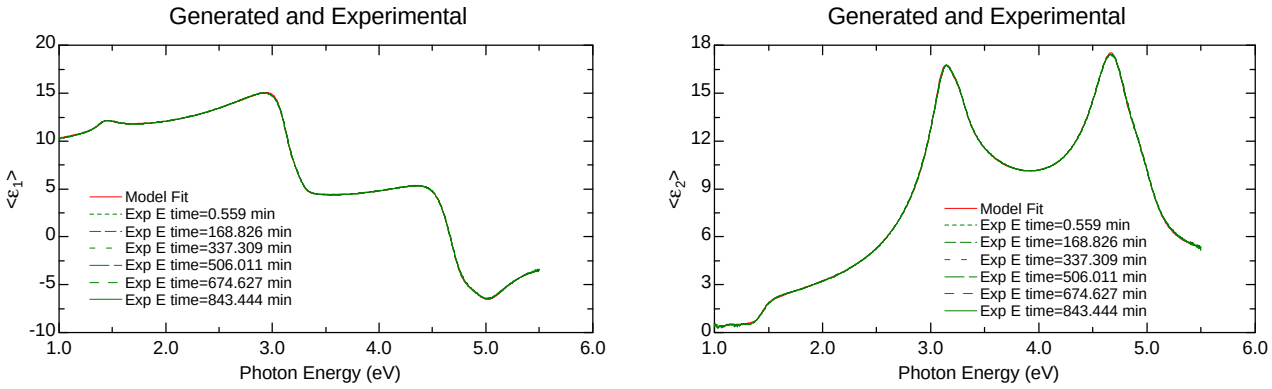

Figure 68a-b. Sulfur doped InP near 297K.

Each measurement cycle appears in the legend denoted by its grouping time.

If dynamic data already exists in the experimental window when a new dynamic scan is started, the new data can be appended to the old data thereby preserving the measurement time information.

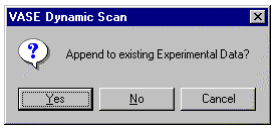

Figure 69

Alternatively, even if the new data will not be appended to the old data, the start time from a previous dynamic scan can still be used. By default, WVASE32® assumes the user will want to maintain a continuous measurement time reference. Answering 'Yes' in the following dialog box resets the dynamic time reference back to zero.

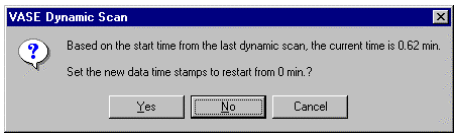
VASE Dynamic Scan
Based on the start time from the last dynamic scan, the current time is 0.62 min.
Set the new data time stamps to restart from 0 min.?
Yes
No
Cancel

Figure 70

In the examples described in the next sections, many scans were started and saved as different files, but the time reference from the first dynamic scan was retained.

Monitoring Sample Cool Down With Temperature Cycling

This sections presents an acquisition and analysis example of thermal cycling to reduce adsorption by the sample surface at very cold temperatures. The sample was a sulfur doped InP wafer. (Room temperature data were previously shown in *Figure 67-Figure 68*.) The dynamic scan used a single wavelength, 427.6nm (2.9 eV) and a 72° angle of incidence. A 50 revolution, zone-averaged (Pol=±20°) acquisition was used. The TempReader.exe utility was put in a one second polling mode while the LakeShore controller units were set to °K.

The data shown in *Figure 71* encompass three cool down cycles and two intermediate warm ups. The User1 values are thermocouple temperatures in °K.

The User1 values will be the secondary Y-axis values "Double-Y axis" is set using Graph|Defaults.

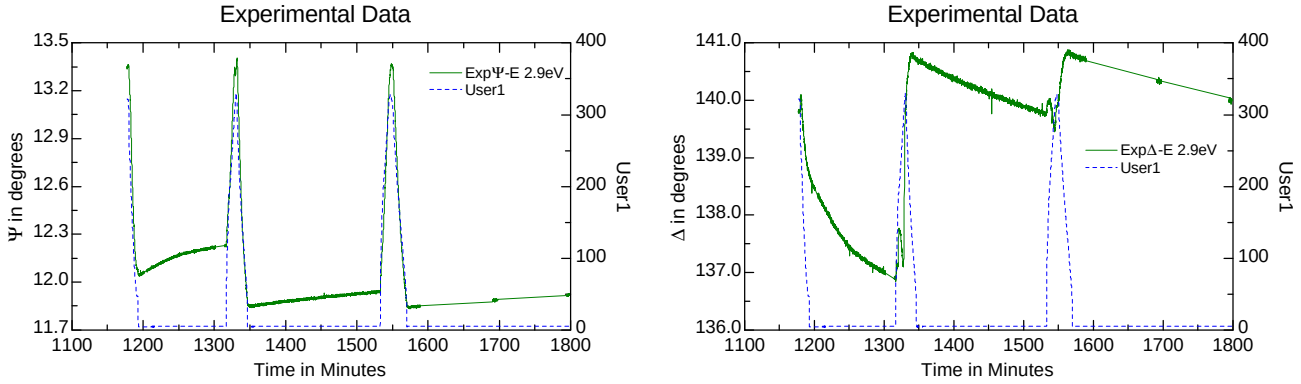

Figure 71a-b. Thermal cycling of sulfur doped InP. User1 is thermocouple temperature.

Cycle #1 (1190-1310 min.) is qualitatively different from cycles #2 (1310-1520 min.) and #3 (1520-1800min.). Qualitatively comparing cycle #1 to #2 and #3, it is noted for cycle #1 that the starting ψ and Δ values are different, that the initial rate of change of the data is greater, and that there is more curvature. These differences are due to the total amount of adsorbed material and the current rate of adsorption. The data during the two warm up cycles exhibit competing effects due to both temperature changes and desorption from the surface.

The key qualitative observation from *Figure 71* is that the sample changes at low temperatures less rapidly for each subsequent temperature cycle. A quantitative analysis of the adsorption rate is given in the next section.

The following procedures are suggested for thermally cycling the sample to reduce the adsorption rate. It is suggested that data be acquired during these steps in the manner described above. The following example assumes liquid helium is used.

Note, the suggested dwell times in the cold phase for cycles #1 and #2 are much shorter than those shown in *Figure 71*. Those measurements were primarily for demonstration of the effect.

The suggested schedule for cycle #1 is as follows:

1. Using the proper procedures described in earlier sections, open the vacuum valves and pump the cyrostat down to 10^{-6}.
2. Adjust the temperature controller set point to 50 K and turn on the heater.
3. Using the proper procedures described in earlier sections, initiate cryogen flow.
4. As temperature approaches 50 K, throttle the cryogen flow back to about ½ turn open on the transfer line bayonet valve. Let the heater try and hold the temperature around 50 K for about 2 minutes. Often there is a large cryostat pressure drop between 50 and 4K, and the goal of this pause at 50 K is to let surfaces other than the sample to also get cold. In this way, hopefully less material will be adsorbed on to the sample.

5. Close the cryostat vacuum valve. By this time, the cryostat will probably be cryo-pumped to a pressure below the turbo pump, and there is no need to cryo-pump gas back streaming from the turbo pump.
6. Turn the heater off and let the temperature fall to 4K.
7. Let the cold-finger assembly and radiation shield soak at 4K for 10 minutes.
8. The initial cycle is complete.

The radiation shield and the whole of the cold-finger assembly should now be cold and participating in cryo-pumping the cryostat chamber. Cycles #2 and #3 quickly ramp the sample temperature back above room temperature. The goal is to clean the sample surface and transfer the adsorbed material to the still-cold radiation shield. The suggested schedule for cycles #2 and #3 are as follows:

1. Stop the cryogen flow.
2. Open the cryostat vacuum valve. There will be a pulse of pressure as the heater turns on in the next step.
3. Adjust the temperature controller set point to 305 K and turn on the heater.
4. After the temperature passes 300 K, wait one minute. The material on the sample surface does not desorb instantaneously. However, do not wait too long or the radiation shield will start to warm up.
5. Turn off the heater.
6. Restart cryogen flow. The transfer line should still be cold, so this time cryogen flow should start almost immediately.
7. As the temperature approaches 50K close the cryostat vacuum valve.
8. Hold at 4 K for 5 minutes.
9. If on cycle #2, repeat steps for cycle #3.

Additional cycles might have some benefit, but they are expected to be minimal. Cycle #3 may be omitted if desired. But as shown in the next section, there is in fact a measurable reduction in adsorption rate with the extra cycle in this experiment.

Adsorption-Rate Data Analysis

At very cold temperatures, the sample under test will adsorb residual gas from the chamber. In a UHV sealed chamber with base pressures below 10^{-4} most of the residual gas is water vapor which is slowly released from the inside surfaces of the cryostat. Because water vapor is the primary contaminant, it is useful to keep the cryostat sealed and under vacuum when not in use and it is useful to minimize exposure time of the cryostat parts to room air when changing samples.

The primary consequence of adsorbing material is that a thin overlayer will grow on the sample during the measurement procedure. Ellipsometry has the wonderful capability of being sensitive to very thin overlayers when the overlayer is the subject of study. However, ellipsometry retains that sensitivity to overlayers even when the overlayers are just complications.

Overlayers themselves are a universal issue when dealing with ellipsometric data. (See “Guide to using WVASE32” for a more in depth discussion of data modeling.) The principal added complication from adsorption is that the overlayer is continually changing. However, that continual change can in turn be used to help characterize

the adsorption layer if certain assumptions about the adsorbant and the adsorption-rate are valid.

The Adsorb layer has been added to the WVASE32® modeling capabilities to deal directly with the adsorption situation encountered with the cryostat. A simple adsorption-rate analysis model is shown below. Layer #1 was derived from a room-temperature analysis of the sample. Layer #2 is just a 'place-holder' layer where the optical constants for the adsorbant material are stored.

3	adsorb (ice)	0 Å
2	ice	0 Å
1	inp-ox	23.985 Å
0	inp_s-doped_tabulated_4k	1 mm

Figure 72

Layer #3 is the 'Adsorb' layer which couples to optical constants from some other layer and defines how the adsorption should be modeled. The 'Adsorb' layer dialog box is shown below.

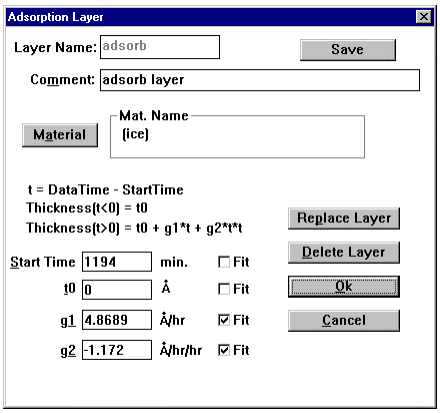

Figure 73

In this example, the optical constants for 'Ice' have been coupled in to the 'Mat. Name' box. Ice optical constants were obtained from a very long adsorption experiment (results not shown here). Future experiments may produce better optical constants, but these optical constants should be satisfactory for most experiments.

The thickness model for the 'Adsorb' layer is summarized in the dialog box. For times before 'Start Time', the thickness is defined as t_0. For times after 'Start Time', the thickness grows as a quadratic polynomial given by Thickness = $t_0 + g_1*t + g_2*t*t$. The rates g_1 and g_2 are give in units of Å/hr and Å/hr/hr respectively. However, note times are in minutes.

Thus the assumptions implicit in the 'Adsorb' layer are that the optical constants are in fact constant for all times and that the layer grows in a manner no more complicated than a quadratic function.

A portion of the data shown in *Figure 71*, is shown analyzed using the model and adsorb layer from *Figure 72* and *Figure 73*. The results are shown below.

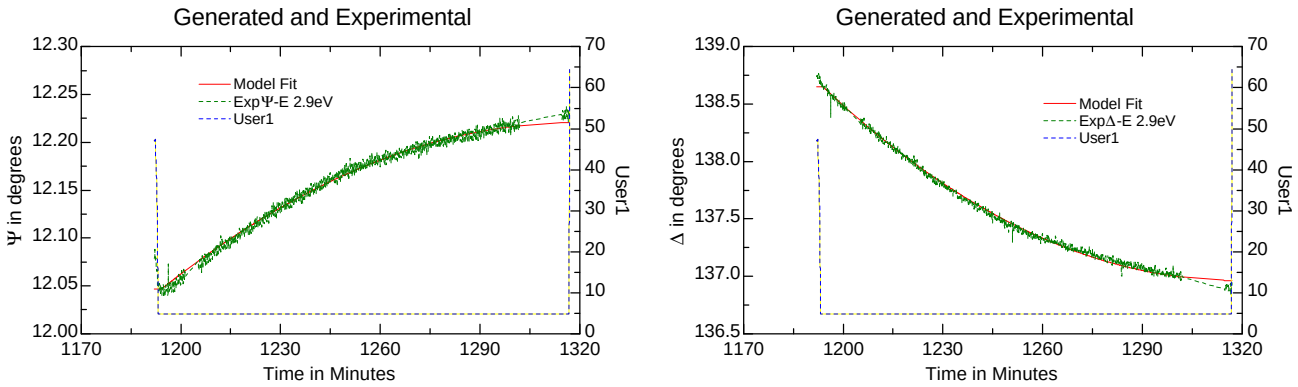

Figure 74a-b. Temperature Cycle #1 fitting for both g_1 and g_2

For the above results, the parameters g_1 and g_2, and the substrate optical constants were fit. Leaving the oxide layer with room temperature thickness and optical constants is the most practical (perhaps only) method of dealing with the oxide overlayer. Note that the assumed start time for adsorption was the 1194 minute mark and that 0 Å of adsorbant was assumed present at that time. Any adsorbant present at 1194 min. was effectively subsumed into the substrate optical constants that were fitted. This type of modeling is typical of the virtual substrate approximation commonly used for in situ monitoring of intentional deposition processes. The key feature of a virtual substrate type model is that the growing layer can be very accurately characterized even when the exact underlying model is not perfectly defined. If there is a good ellipsometric measurement a some time (e.g. 1194 min.) and the underlying sample is of the correct type (high index, slightly absorbing is best), the virtual substrate approach is possible.

To examine the impact of the g_2 fit parameter on the analysis, see the following figures.

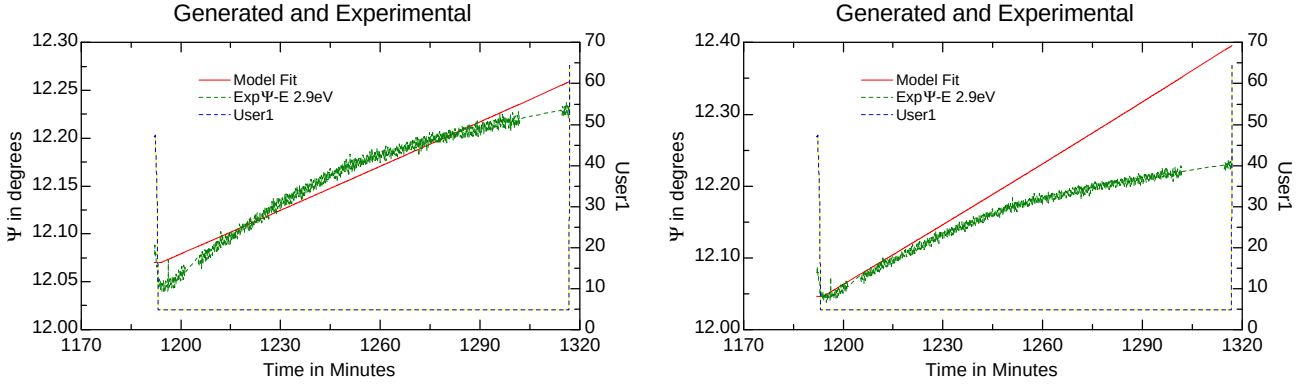

Figure 75a-b. Temperature Cycle #1 with different constant rate model.

In *Figure 75a*, the same fitting analysis was performed except that the constraint g_2=0 was enforced. A constant adsorption rate is not the best model. In *Figure 75b*, the g_2 from the original model was reset to zero and data generated assuming no change in adsorption rate occurred.

The last part of this temperature cycle (1290 - 1317 min.) was subjected to a similar analysis using a constant growth rate analysis. The resulting 'Adsorb' layer and data fits are shown in the following Figures.

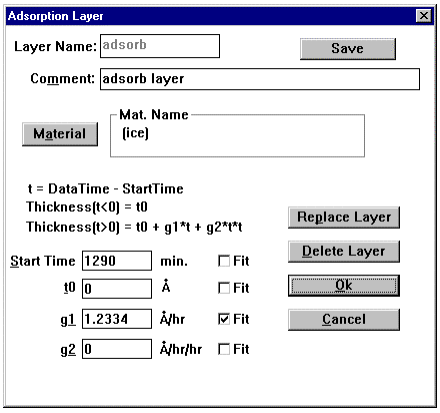

Figure 76

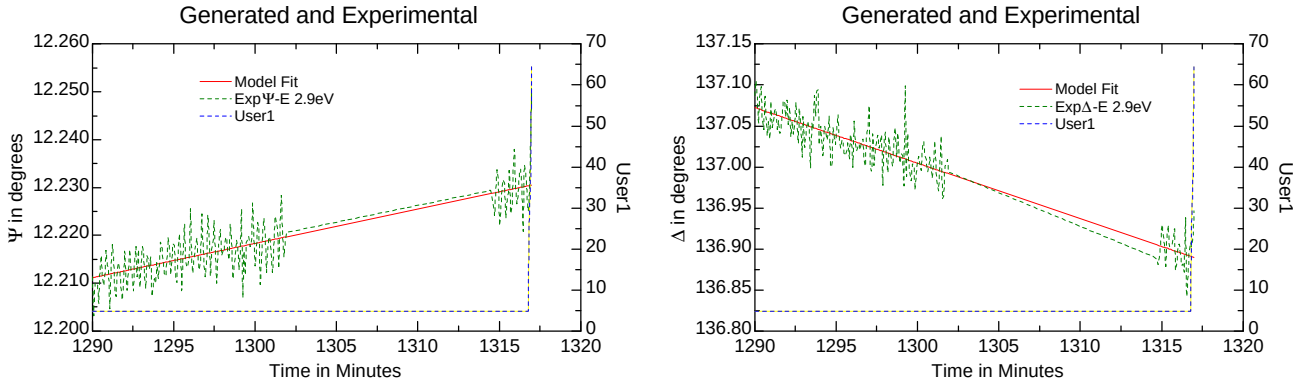

Figure 77a-b. End of Temperature Cycle #1

Over the final 30 minutes of the first temperature cycle, a constant adsorption rate is a satisfactory model. Note, the gap in the data. During this time a quick spectroscopic scan (not shown) was taken. When the single-wavelength monitoring resumed, the VASE-dynamic time reference was maintained so a continuous data set would be available for analysis.

Cycle #2 was subjected to a similar adsorption-rate analysis with results shown below.

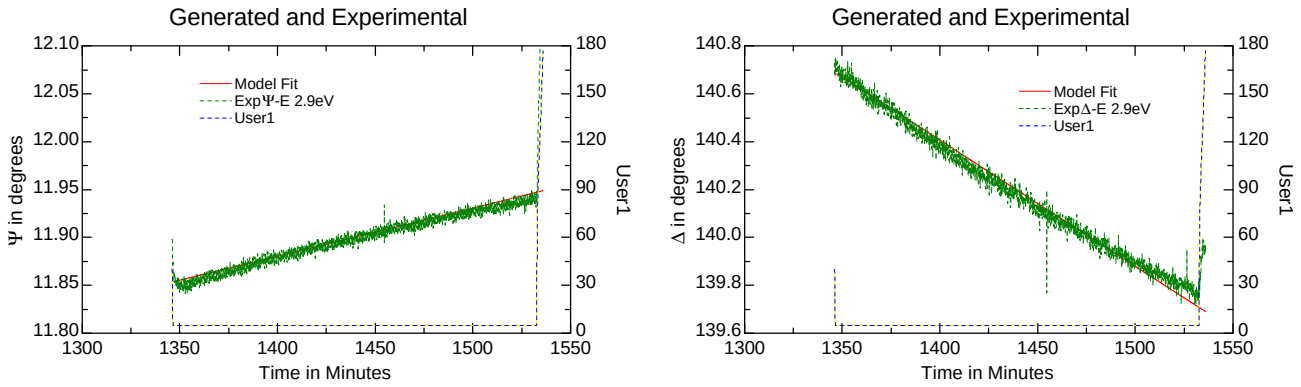

Figure 78a-b. Temperature Cycle #2

For cycle #2, a constant adsorption-rate was sufficient for the entire cycle time. Also, note the starting ψ and Δ values from cycle #2 were not the same as cycle #1. A reasonable explanation is that during the cool down of cycle #1, the sample adsorbed a non-negligible amount of ice which was present at the 1194 min. mark.

This implies that 1) the previous model assumption that the adsorb layer had zero thickness at 1194 min. was probably wrong. (But remember, this affects only the fitted-for optical constants of the substrate, not the adsorption-rate analysis.) And therfore 2) the fitted substrate optical constants from cycle #2 are closer to the actual 4 K optical constants of this sample.

For cycle #3, a constant adsorption-rate was also sufficient to model the entire 4 K data region. Note, most of the time was spent acquiring spectroscopic data (see next section). However, it was useful to take single-wavelength data at the beginning and the end to determine the adsorption rate (used in the next section) and to confirm that a constant adsorption-rate is the correct model.

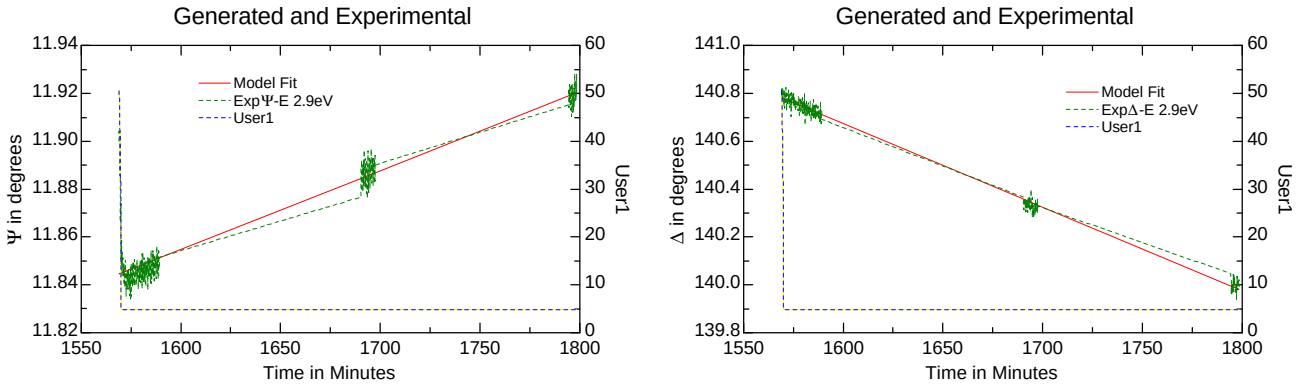

Figure 79a-b. Temperature Cycle #3

The results from Cycles #2 and #3 are very similar. The starting ellipsometric values are very similar suggesting that no further cleaning was achieved by the second warm up step, and suggesting that the surface is probably free of most adsorbant when the 4 K temperature was reached. There is a slight reduction (improvement) in the rate of adsorption for Cycle #3. The following table summarizes the adsorption rate for different sections of the temperature cycling experiment.

Table 1 Adsorption-Rate Analysis Results

Cycle #1, start	4.869 Å/hr
Cycle #1, end	1.233 Å/hr
Cycle #2	0.919 Å/hr
Cycle #3	0.606 Å/hr

Low-Temperature Spectroscopic Measurement

Low temperature spectroscopic measurements were taken during temperature cycle #3 for the sulfur doped InP sample. The same adsorption rate analysis model as used for the data in *Figure 79* was used for the results shown below. For the spectroscopic data however, the adsorption rate was held constant and the InP optical constants were fit over the full range of wavelengths measured instead of just the monitoring wavelength.

In particular, a parametric-semiconductor layer was used to model the substrate optical constants. This model is based on Kramers-Kronig consistent functions with Gaussian broadening. Guassian broadening is needed to correctly model the sharp

direct-gap region for a material like InP especially at low temperatures. Alternatively, the optical constants could have been fit for on a wavelength-by-wavelength basis. However, in many cases, the function-based model is preferred because the physical K-K consistency is built in.

The data and fit results for three wavelength cycles are shown below in *Figure 80* and *Figure 81*. Although not clearly visible due to the scale of these graphs, the data for each wavelength cycle is slightly different because the adsorbing layer continues to grow throughout the acquisition process. That is why each measurement needs to have its own correct measurement time recorded. The grouping time is only needed to ease graphing and experimental data selection. Note, as discussed in "Guide to Using WVASE32", the pseudo-dielectric values $\langle\varepsilon_1\rangle$ and $\langle\varepsilon_2\rangle$ in *Figure 81* are just numerically transformed from the ψ and Δ values.

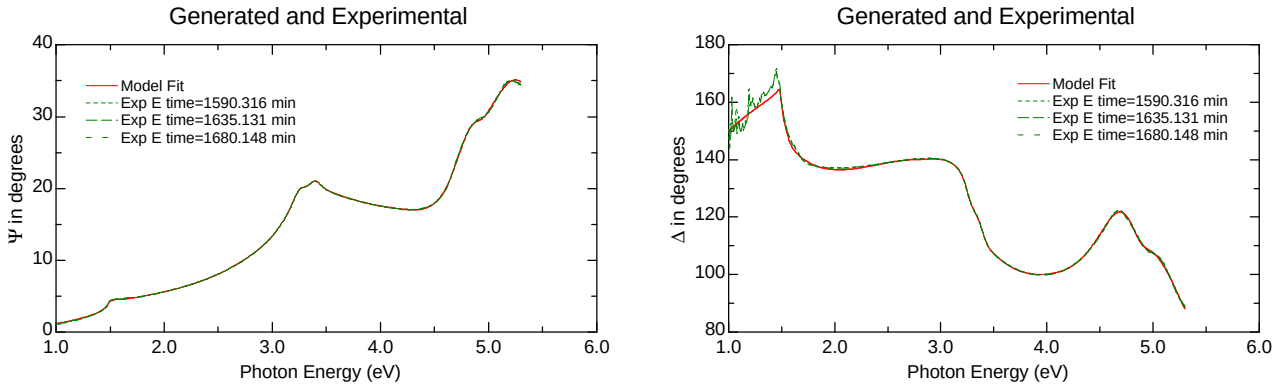

Figure 80a-b. Sulfur doped InP near 4K.

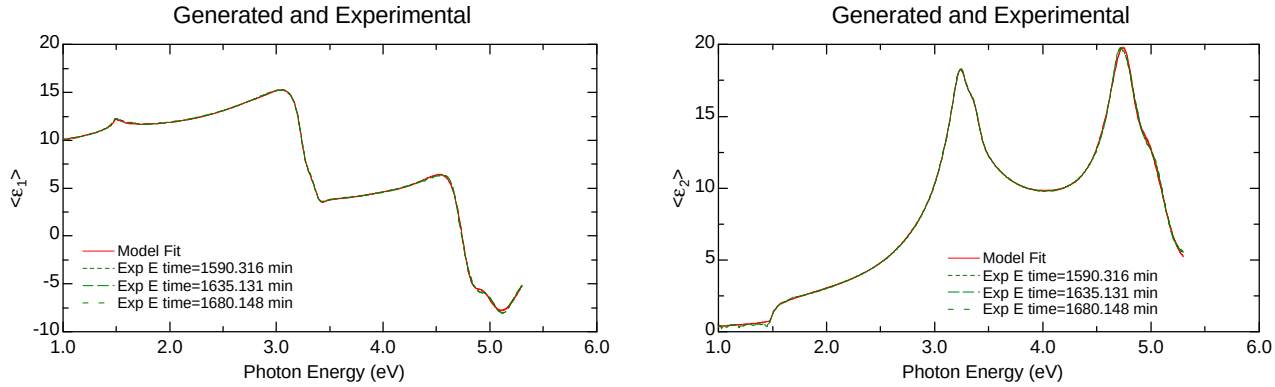

Figure 81a-b. Sulfur doped InP near 4K.

The room temperature data displayed in *Figure 67* and *Figure 68* was subjected to a similar parametric-semiconductor model analysis. Of course, there was no adsorbing layer to correct for. A comparison of room-temperature and near-4K data is shown in the figures below. Note how the critical point structures sharpen and shift for 4K as compared to 297K.

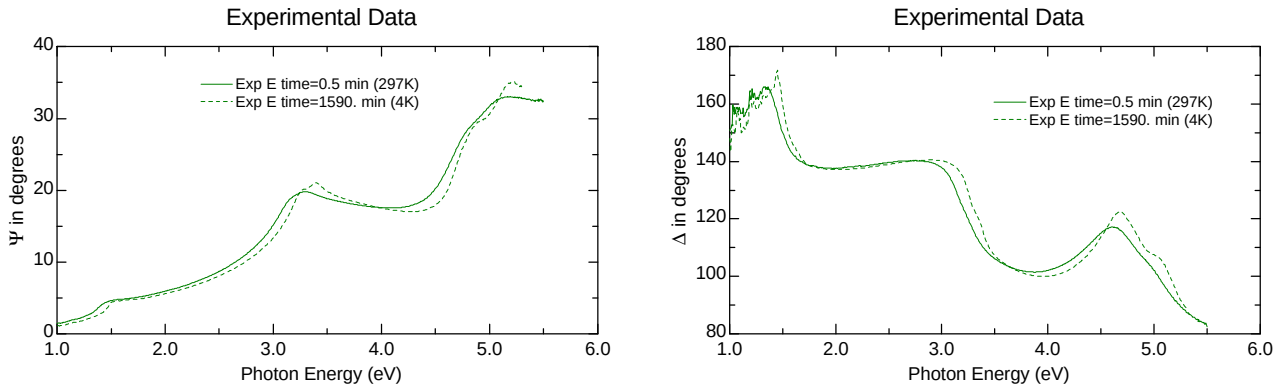

Figure 82a-b. Sulfur doped InP: Comparison of 4K and 297K data.

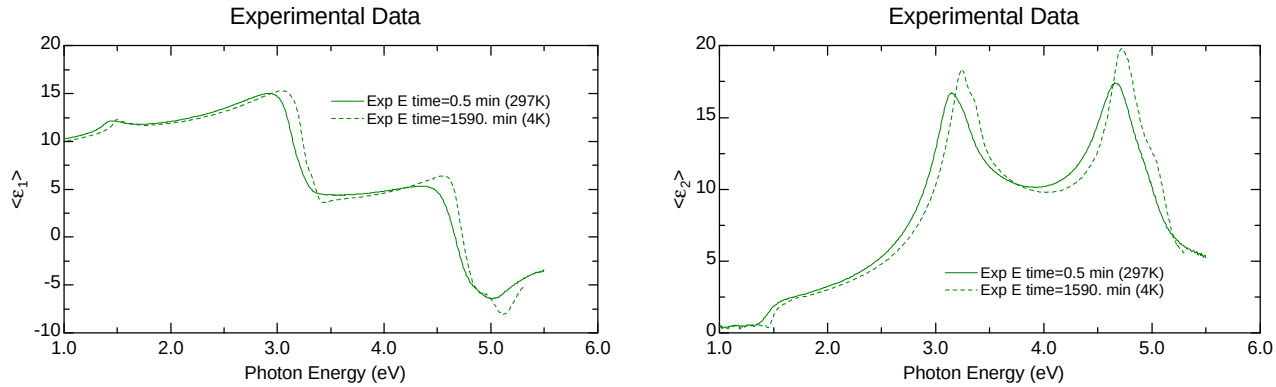

Figure 83a-b. Sulfur doped InP: Comparison of 4K and 297K data.

High-Temperature Spectroscopic Measurement

A few comments about high-temperature operation of the cryostat are needed before some experimental results for a native-oxide silicon sample are shown.

- Remove the transfer line bayonet from the cryostat, before making high-temperature measurements in the cryostat. The transfer line bayonet may be damaged if exposed to temperatures above 475K.
- The cryostat should be under vacuum to prevent corrosion of the internal components which might occur if exposed to large oxygen concentrations at high temperatures.

Be careful! Some materials are capable of out gassing toxic chemicals at high temperatures

- Some materials are capable of out gassing toxic chemicals at high temperatures (e.g. GaAs may out gas As). Be careful, and consider what is going to happen to your sample at high temperatures under vacuum. Coating windows and interior surfaces may affect future operation, and the problems are complicated many times over if the contaminant is toxic.
- The surface morphology of some materials may be altered at high temperature. Furthermore these changes are likely to be irreversible. The example data presented below gives a good example of how this can happen.

The following experimental data was taken for a native-oxide on silicon sample. The single-wavelength tracking data was acquired at 3.1 eV. At this wavelength the silicon optical constants exhibit a strong dependence on temperature.

It is best to start with lower temperatures and the move to higher temperatures. At high temps the sample may be irreversibly changed.

The data was not subjected to a detailed analysis, because the sample surface was irreversible altered at temperatures above 600 K. In fact, this data provides an excellent example of how NOT to perform high temperature measurements. As a general rule, perform room temperature measurements first. Next, perform the low-temperature measurements of interest. Finally perform the high-temperature measurements of interest working from the lowest to the highest temperature.

The data at 3.1 eV is summarized in *Figure 84* for an extended high-temperature run. The experiment started by ramping the sample up to 650 K. As previously mentioned, a better procedure would have been to start at 400 K and work up to 650 K. An important feature in the data can be observed in first 200 minutes where the ellipsometric measurements show a continuous change even after the temperature was fully stabilized. This was indicative of the surface morphology changing. This is similar to the situation at low-temperatures where the growing adsorption layer change the ellipsometric data. However, this high temperature change was irreversible. This change was confirmed by noting the change in room-temperature measurements from before and from after this experiment (not shown).

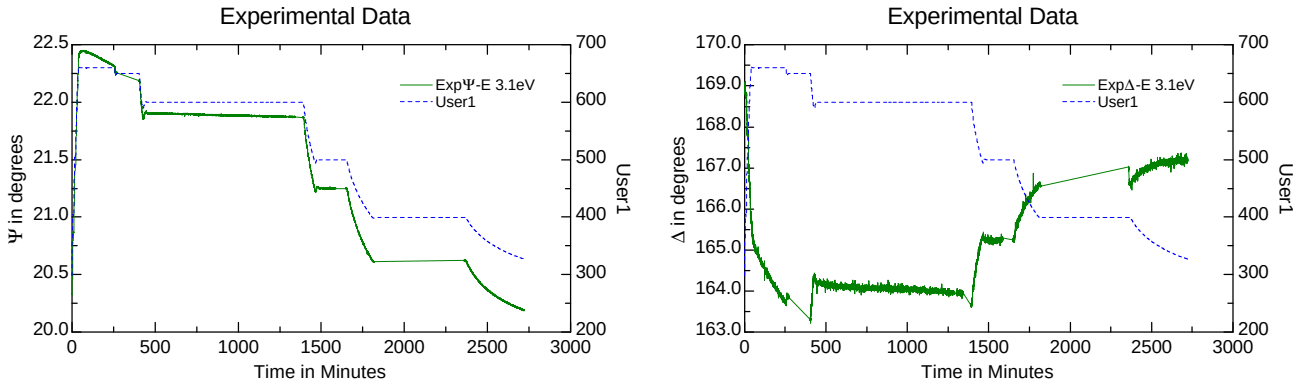

Figure 84a-b. Native oxide silicon wafer at elevated temperatures.

More detailed looks at the data are shown in *Figure 85*. The slope in ψ with constant temperature is easily seen for temperatures above 600K. Also, note the undershoot of the temperature as the control point was changed. Especially note the 2-3 minute lag of sample temperature (as seen in the ψ) with respect to the thermocouple in *Figure 85b*.

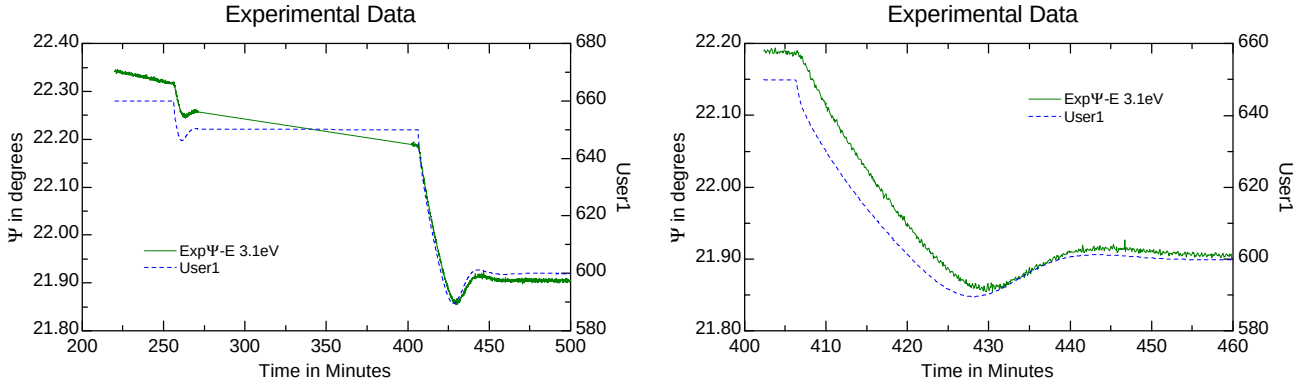

Figure 85a-b. Native oxide silicon wafer at elevated temperatures.

After the high temperature measurements, the same sample was measured at low temperatures. A comparison of spectroscopic data for three different temperatures is shown in *Figure 86* and *Figure 87*. The dominant features are due to the silicon substrate. The surface overlayer, although modified by the high-temperature experiments is still thin ~30Å. Thus although the data is sub-optimal for the accurate determination of silicon optical constant, the key features (energy shifts and broadening changes) of the temperature dependence are clearly present.

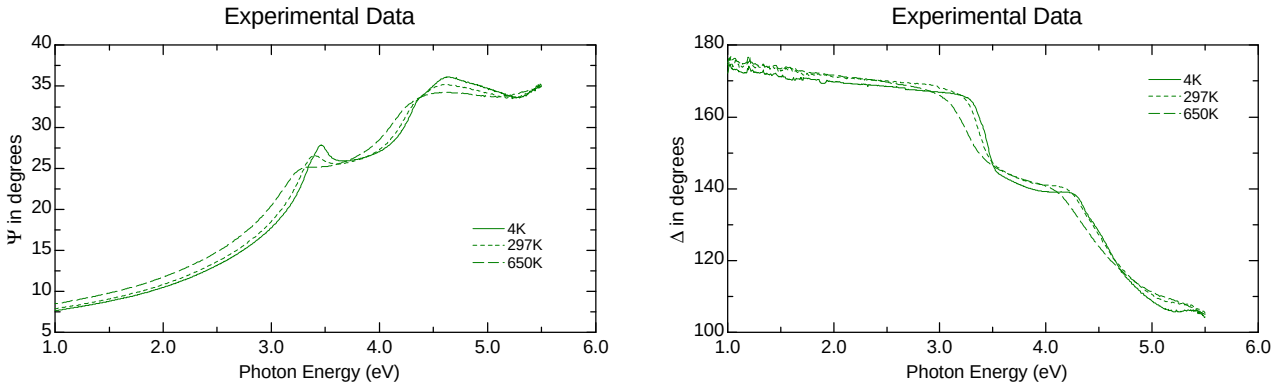

Figure 86a-b. Native oxide silicon wafer from 4K to 650K.

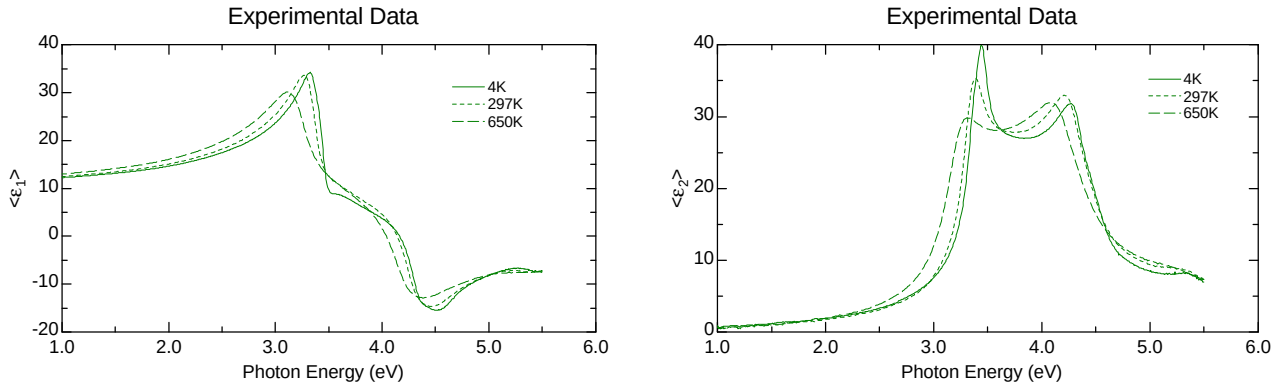

Figure 87a-b. Native oxide silicon wafer from 4K to 650K.

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